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Professional Paper 165—A

LITHOLOGIC STUDIES OF FINE-GRAINED UPPER CRETACEOUS
SEDIMENTARY ROCKS OF THE BLACK HILLS REGION

BY

WILLIAM W. RUBEY

Shorter contributions to general geology, 1930

(Pages 1-54)



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SHORTER CONTRIBUTIONS TO GENERAL GEOLOGY, 1930

LITHOLOGIC STUDIES OF FINE-GRAINED UPPER CRETACEOUS SEDIMENTARY ROCKS OF THE BLACK HILLS REGION

By WILLIAM W. RUBEY

ABSTRACT

More than nine-tenths of the Upper Cretaceous rocks in northeastern Wyoming are fine-grained shales, mudstones, and calcareous marls. A comparative study of the mineralogy, chemical and mechanical composition, density and porosity, fissility, and lamination of samples of these rocks discloses several relations that throw light on the geologic history and structural deformation of the region, and perhaps on its oil and gas possibilities.

Microscopic examination of thin sections and rock powders shows that platy crystals of a clay mineral (probably beidelite) form the chief constituent of the argillaceous rocks. Most of these platy crystals lie essentially parallel to the bedding and give the rocks an aggregate optical orientation. Calcium carbonate occurs in varying amounts in many of the samples as shells, crystals, microcrystalline lenses, and spherulites. Opaque organic matter, fine quartz sand or silt, and small nodules of pyrite are minor constituents of most of the samples. A pyritic limestone 4 feet thick from the Greenhorn formation consists of calcite, pyrite (and either pyrrhotite or ferrous sulphide), and gypsum, with smaller amounts of iron oxide, organic matter, and bone phosphate. The gypsum in this limestone is secondary, but the sulphide, which makes up one-fourth of the rock, is probably almost, if not quite, syngenetic. Field relations indicate that the sedimentation by which this pyritic limestone was formed was not unusually slow.

Partial chemical analyses indicate that the black shales contain less organic matter than the light-colored calcareous marls and that there is less chloroform-soluble bitumen in the formations of Benton age than in the overlying Niobrara and Pierre formations. The proportion of chloroform-soluble bitumen varies also, with either the dip or the porosity of the rocks; it is not possible to say which. This indicated relationship to the dip of the rocks is of economic interest, for it suggests the possibility that deformation has converted organic matter into petroleum, and that undrilled areas of steep dips and faulting may contain oil. In general, the percentages of calcium carbonate, organic matter, and pyrite vary together; the older formations contain least, the Pierre shale more, and the Niobrara and Greenhorn formations most. A consideration of the conditions favorable for the formation and preservation of these three constituents and of the abundance of remains of bottom-living mollusks in rocks near one end of this series and the absence of fossils in rocks near the other end, together with other facts, leads to the conclusion that the more calcareous, organic, and pyritic sediments probably accumulated more rapidly and in shallower water than the others. In fact, the entire series of sediments seems to be very similar to the blue muds accumulating near present coasts,

In choosing a method for determining the mechanical composition of these fine-grained rocks, many methods used in other sciences were reviewed. A mechanical analysis consists of three parts—preparation of the samples, measurement, and presentation of results. The attainable accuracy of each step in all parts of the analysis, as well as the uncertain relation of the present to the original size of the particles, must be considered in choosing the particular methods best adapted for the purpose in view. Before most fine-grained sedimentary rocks can be mechanically analyzed they must be disintegrated by chemical treatment and mechanical agitation. In this paper different methods of preparation are discussed, and a long period of soaking in water is suggested as an additional treatment. The samples may be measured by sieving, microscopic counts, or settling in a fluid. The methods that depend upon the different settling velocities of large and small particles include elutriation, decantation, increasing weight of fallen sediment, and decreasing density or turbidity of the suspension. The merits and demerits of many modifications of the different methods are considered, and the conclusion is reached that some methods give much more information in less time than others. Pyramidal diagrams and cumulative curves, plotted logarithmically, seem to present the results of measurement most satisfactorily; but the conversion of the data into these diagrams presents a different problem for each method of measurement. A simple graphic solution saves much work in converting the data obtained by many settling or "sedimentation" methods.

As a result of this review, rock samples and insoluble residues of some calcareous marls were disintegrated by two months' soaking in slightly ammoniacal water, with occasional shaking and rubbing. Organic matter retarded disintegration and dissolved slowly. The samples were measured by frequent weighing of the sediment that accumulated on a pan which hung in a thoroughly mixed suspension. An attempt to use an aluminum pan proved unsuccessful, and a platinum pan was substituted. The unexpectedly high porosity of the settled sediment caused some difficulties. It was found that the weight of sediment increased as the logarithm of the settling time and that sufficiently accurate estimates of the final weight could be made from a series of successive determinations of weight made in less than one hour. Somewhat less sediment accumulated on the pan than was expected from the size of the original sample, but investigation indicated that this loss was due chiefly to solution of organic matter, interstitial water, and fine particles, and to slumping of sediment off the edges of the pan. The successive determinations of weight and settling time were corrected for admixed small particles and the temperature of the water and thus converted into percentages and settling velocities. The exact relation between settling velocities and diameters of irregular particles is unknown, and examination of samples pipetted off during set-

ting suggested that the flat shape and large surface area of the clay minerals may be the chief source of this uncertainty. Similarly, the size terms "sand," "silt," and "clay" are not defined by diameter limits that are universally accepted. Therefore, the settling velocities were not recomputed into theoretical diameters but were plotted directly alongside the corresponding size terms, which can be correlated arbitrarily with settling velocities by a study of published measurements. Several methods of checking indicated that the final graphs are accurate within about 2 to 5 per cent. These graphs show that the samples consist almost entirely of grains smaller than very fine sand and that they are essentially unsorted mixtures of silt and clay. If this lack of sorting is due to flocculation of the particles at the time of deposition, it may be a criterion of origin in saline or marine water. The grains of calcium carbonate in the more calcareous samples seem to be better sorted than the associated detrital material.

The density and porosity of these fine-grained rocks were found to vary greatly with the temperature to which the samples had been heated. Below 200° C. this change is due almost entirely to loss of water, and consequently all determinations were made on samples previously heated to this temperature. The grain or mineral density varies with the impurities in the rock and with the size of the grains. The lump or rock density is about 2.0 or less, indicating that the positive anomalies found at some gravity stations in the Black Hills region are not caused by unusually heavy rocks near the surface. The porosity averages about 30 per cent, and it decreases apparently by quantitative relationships with increase in the dip of the beds and in the original depth of burial. The size of grain seems to be another factor affecting porosity, the finer-grained samples having been most compacted. The relation of porosity to dip and to proximity of faults is significant, for it indicates that these rocks were deformed internally by horizontal compression, that their apparent stratigraphic thickness depends upon the degree of folding, and that large volumes of water were squeezed out of them during deformation.

Field evidence, such as the greater abundance of fissile shales in stratigraphically lower beds and the slight discordances between fissility and bedding noted here and there, suggests that the fissility or shaly structure is secondary. Microscopic examination and chemical and mechanical analyses neither confirm nor refute this suggestion. Some microscopic cracks that may represent planes of fissility are slightly inclined to the bedding, but the aggregate optical orientation (probably incipient fissility) is essentially parallel to the bedding. In the samples studied fissility varied inversely with the content of calcium carbonate but was unrelated to the size of the grains.

Many of the samples exhibit more or less distinct laminations of several types that may be annual layers. The climate and other physical conditions were probably favorable for the formation and preservation of annual layers in these rocks. The average thickness of pairs of the laminations is roughly the same as the observed thickness of annual layers deposited elsewhere and the expected thickness of annual layers in these rocks as estimated by several independent methods. More detailed comparison with the probable rate of transgression of the Upper Cretaceous sea and with the supposed length of Upper Cretaceous time suggests that each pair of laminations may represent several years' deposits, but these discrepancies may be the result of many undetected discontinuities or diastems in the stratigraphic sequence. The distinctness and thickness of the laminations in the different samples indicate that the shales accumulated slowly in rather

deep water and that the sandier rocks and calcareous marls accumulated more rapidly and in shallower water. The discussion of these bedding laminations is summarized more fully in the report (pp. 52-53).

Three of the broader conclusions are based upon scattered evidence. The chemical composition, fossil content, bedding laminations, lack of sorting, and comparison with present-day blue muds indicate that, in general, the finer-grained non-calcareous shales accumulated more slowly and in deeper water than the calcareous, organic, and pyritic rocks. The variations in the percentage of pore space and soluble bitumens, in the fissility and aggregate optical orientation, and in the thickness of formations indicate that these fine-grained rocks lost volume and were deformed internally by loading and tilting. The percentage of soluble bitumens, probably a measure of the oil content, seems to vary with the tilt or shear to which the rocks have been subjected but not with the depth or load. Although this relation may be fortuitous, it suggests the possibility that folding may convert the organic matter in fine-grained sedimentary rocks into oil and then force it into adjacent sandstone beds.

INTRODUCTION

Fine-grained argillaceous rocks constitute the most abundant type of sedimentary rocks, and their peculiar properties make them of special interest in geologic studies. The conditions of their deposition are very different from those of the coarser-grained sandstones; they yield to deformation by compaction and plastic distortion in a manner quite unlike the coarser rocks; and, to mention only one example of their economic importance, they are commonly thought to be the source beds of petroleum. Consequently, from both the scientific and the practical points of view, they deserve careful study. Yet, though they have been widely studied and the literature describing them is voluminous, they probably are the least understood of the sedimentary rocks. This perplexity is due in part to the difficulty of studying them microscopically, owing to their incoherence and the small size of their constituent particles, and in part to their extremely diverse chemical composition. However, recent advances in microscopic technique and in the mineralogy of clays and improved methods of chemical and physical examination give promise of placing the study of argillaceous rocks on a much more satisfactory basis.

More than nine-tenths of the Upper Cretaceous rocks exposed on the northwest flank of the Black Hills in Wyoming and Montana are shales, mudstones, and calcareous marls. Consequently, in a somewhat detailed study¹ of these Upper Cretaceous rocks, with special reference to their geologic history, types of structural deformation, and oil and gas possibilities,

¹ Rubey, W. W., Origin of the siliceous Mowry shale of the Black Hills region: U. S. Geol. Survey Prof. Paper 154, pp. 153-170, 1929; Cretaceous and Cenozoic formations on the northwest flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [in preparation]; The oil and gas possibilities of the Black Hills rim in Wyoming and Montana: U. S. Geol. Survey Bull. — [in preparation].

it became necessary to devote particular attention to these fine-grained sedimentary rocks.

The present report gives the results of a comparative study of some of the outstanding lithologic characteristics of a group of samples chosen as representative of the rock types in the different formations of this region. Except for the theoretical discussion of the bedding laminations, this paper is essentially an empirical or descriptive study of the lime-clay series of sedimentary rocks and an attempt to interpret the observed relations between the characteristics of the different rock types. The conclusions reached are merely tentative. The relationships pointed out and the explanations offered could be definitely substantiated only by a much more detailed study of the rocks within this area and by observations extending over a much wider area. However, if any part of the paper suggests fruitful problems in this or other areas or proves helpful to others who are studying similar rocks, it will have accomplished its chief purpose.

The writer is indebted to his colleagues, most of them members of the United States Geological Survey, for many helpful criticisms and suggestions in the

preparation of this report. W. H. Bradley contributed to all parts of the paper by his generous discussions; C. S. Ross gave advice on the mineralogy of the rocks; the Bureau of Mines kindly made the determinations of organic matter and Taisia Stadnichenko offered valuable suggestions in their interpretation; C. K. Wentworth reviewed the section on mechanical analyses; P. G. Nutting determined the densities and porosities and helped in their interpretation; and Adolph Knopf and J. B. Reeside, jr., criticized the discussion of bedding laminations.

ROCKS STUDIED

The stratigraphic relations and areal extent of the Upper Cretaceous rocks that crop out for about 150 miles along the northwest flank of the Black Hills uplift are described in another report.² Their general characteristics and thickness, together with the stratigraphic names adopted in that report, are summarized in the following table:

² Rubey, W. W., Cretaceous and Cenozoic formations on the northwest flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [in preparation].

Upper Cretaceous formations in northeastern Wyoming and southeastern Montana

Series	Group	Formation and member	Thickness (feet)	General characteristics	
Eocene.		Wasatch formation.	100+	Continental deposits.	
		Fort Union formation.	Tongue River member.		650-1,050
			Lebo shale member.		
Eocene (?).		Lance formation.	Tullock member.	1,000-2,250	
			Hell Creek member.		
Upper Cretaceous.	Montana.	Fox Hills sandstone.	150-250	Brownish sandy shale and siltstone with beds of sandstone and ferruginous concretions. Marine fossils. Gradational into underlying Pierre shale. Forms a prominent grassy scarp.	
		Pierre shale.		150-250	Dark-gray fissile shale and mudstone with calcareous concretions. Locally contains light-buff sandy shale. Marine fossils.
			Monument Hill bentonitic member.	150±	Impure bentonite and siltstone. Some calcareous and barite concretions. Marine fossils. Commonly forms a scarp. Named from exposures at Monument Hill, sec. 32, T. 56 N., R. 68 W., Crook County, Wyo.
				500-800	Dark mudstone and shale with abundant calcareous concretions. Light gray in upper part; iron-stained in lower part. Marine fossils.
			Mitten black shale member.	150-200	Blue-black fissile shale with few iron-stained calcareous concretions. Marine fossils. Forms a prominent scarp. Named from exposures along Mitten Prong in T. 56 N., R. 68 W., Crook County, Wyo.

Upper Cretaceous formations in northeastern Wyoming and southeastern Montana—Continued

Series	Group	Formation and member	Thickness (feet)	General characteristics	
Upper Cretaceous.	Montana.	Pierre shale. Gammon ferruginous member. Groat sandstone bed.	800-1,000	Abundant iron-stained concretions and thin beds of siderite in light-gray mudstone and shale. Fossils scarce but consist of marine species. Commonly forms bare buttes. Named from exposures along Gammon Creek in T. 57 N., Rs. 67 and 68 W., Crook County, Wyo. Groat sandstone bed near top of member (150 feet thick in northern part of area) consists of ferruginous and glauconitic sandstone and siltstone. Named from exposures along Groat Creek in T. 7 S., R. 56 E., Carter County, Mont. Pedro bentonite bed at base of member (locally 20 feet thick but not widespread) consists of hard white massive clay and tuff. Named from exposures near Pedro, sec. 5, T. 45 N., R. 63 W., Weston County, Wyo. Possible unconformity at or near base of member.	
	Colorado.	Niobrara formation.	Beaver Creek chalky member.	125-200	Chalk marl and calcareous siltstone, gray where fresh but weathering to light yellow. Marine fossils. Named from exposures along Beaver Creek in T. 46 N., R. 64 W., Weston County, Wyo.
			Sage Breaks shale member.	250-325	Gray noncalcareous mudstone and shale with many large light-gray calcareous septarian concretions. Fossils scarce but consist of marine species. Commonly forms scarps and buttes. Named from exposures in the Sage Breaks, in T. 46 N., R. 63 W., Weston County, Wyo. Included in Carlile of previous reports.
		Carlile shale.	Turner sandy member.	150-200	More or less sandy shale and siltstone with iron-stained concretions. Persistent thin beds of sandstone, locally conglomeratic and phosphatic and containing abundant shark teeth, in lower part. Marine fossils. Forms a minor scarp. A distinct faunal break and possible unconformity at base. Named from exposures along Turner Creek in Tps. 46 and 47 N., R. 64 W., Weston County, Wyo.
				75-125	Dark-gray shale with a few calcareous concretions. Marine fossils.
		Greenhorn formation.	50-350	Chalk marl, thin-bedded limestone, and light-gray sandy shale with calcareous concretions. Marine fossils. Forms a prominent scarp. Interfingers with underlying Belle Fourche shale. Limestone facies in south; thin concretionary facies in northwest; thick chalk-marl facies in northeast.	
		Graneros shale.	Belle Fourche shale member.	350-1,000	Black fissile shale and mudstone with concretions and bentonite beds in upper half and lowermost part. Upper concretions calcareous; lower ones sideritic. Fossils scarce in lower part, but all are marine species.
	Mowry siliceous shale member.		125-225	Hard siliceous claystone, dark gray where fresh, light silvery gray where weathered. Contains many fish scales. Many thin beds of bentonite. Marine fossils. Forms a scarp or ridge. Gradational into and includes at base a few feet of soft shale ("Nefsy shale member"). Clay Spur bentonite bed (1 to 4 feet thick) at top over entire area. Named from exposures near Clay Spur, sec. 30, T. 47 N., R. 63 W., Weston County, Wyo.	

Upper Cretaceous formations in northeastern Wyoming and southeastern Montana—Continued.

Series	Group	Formation and member		Thickness (feet)	General characteristics
Upper Cretaceous.	Colorado.	Graneros shale.	Newcastle sandstone member.	0-75 (commonly 40).	An extremely variable unit of discontinuous beds of sandy shale, sandstone, impure lignite, bentonite, and (where thin) phosphatic nodules. Contains both continental and marine or brackish-water fossils. Local unconformities within member.
			Skull Creek shale member.	175-275	Black fissile shale with few ferruginous concretions. Thin sandy beds in lower part. Fossils scarce, but consist of marine species.
Lower Cretaceous.	Inyan Kara.	Fall River ^a sandstone (=so-called Dakota sandstone of previous reports on this area).	?	150-350	An extremely variable group consisting of discontinuous beds of sandstone, sandy shale, conglomerate, lignite, and variegated siltstone. In general, though not in detail, the higher sandstones are more heavily iron stained and slabby and the lower ones lighter gray and massive. Continental fossils throughout greater part but marine fossils in upper 20 feet. Named from exposures along Inyan Kara Creek in the northeastern part of the Moorcroft quadrangle, Wyo.
Fuson formation.		?			
Lakota sandstone.		?			

^aRussell, W. L., The origin of artesian pressure: *Econ. Geology*, vol. 23, pp. 134-136, 1928.

A group of samples (air-dried from three to four years) was selected for chemical and mechanical analyses and determinations of density and porosity. These samples are described below.

A. Black fissile shale about 50 feet above base of Skull Creek member of Graneros shale. Associated with thin sandy beds and fossiliferous ferruginous concretions. Center of sec. 28, T. 50 N., R. 65 W., Crook County, Wyo. Beds dip 5° SW.

B. Dark-gray hard siliceous claystone from uppermost part of Mowry member of Graneros shale. Associated with bentonite beds and contains many Radiolaria. Center of sec. 35, T. 57 N., R. 66 W., Crook County, Wyo. Beds dip 3° NW.

C. Dark-gray to black shale or mudstone from upper few feet of Belle Fourche member of Graneros shale. W. ½ sec. 34, T. 58 N., R. 62 W., Crook County, Wyo. Beds dip 4° NE.

D. Pyritic limestone from basal bed of Greenhorn formation, a 4-foot bed consisting of 1-inch layers of highly fossiliferous, somewhat sandy limestone. Contains mollusks, ammonites, fish bones, and teeth. Lowermost layer contains the most pyrite. Immediately overlies with sharp contrast shale like sample C. SW. ¼ sec. 11, T. 57 N., R. 62 W., Crook County, Wyo. Beds dip 6° NE.

E. Light-buff to drab calcareous marl about 60 feet below top of Greenhorn formation. Contains many small mollusks. Associated with thin beds of fossiliferous sandy limestone, pyrite nodules, and silty chalkstone concretions. NW. ¼ sec. 14, T. 57 N., R. 62 W., Crook County, Wyo. Beds dip about 1° NE.

F. Dark-gray shale about 15 feet above base of Turner member of Carlile shale. Associated with sandy shale and thin beds of coarse sandstone. W. ½ sec. 19, T. 47 N., R. 64 W., Weston County, Wyoming. Beds dip 33° SW.

G. Gray shale or mudstone from Sage Breaks member of Niobrara formation. Associated with many large calcareous septarian concretions. SW. ¼ sec. 5, T. 45 N., R. 62 W., Weston County, Wyo. Beds dip 50° S.

H. Light-gray calcareous marl (unweathered) from lower part of Beaver Creek member of Niobrara formation. SW. ¼

sec. 30, T. 46 N., R. 63 W., Weston County, Wyo. Beds dip 10° SW. and lie between two small normal faults.

I. Gray siltstone from lower part of Gammon member of Pierre shale. Associated with many thin beds of siderite and iron oxide. Collected near a fossil vertebrate skeleton. S. ½ sec. 32, T. 49 N., R. 66 W., Crook County, Wyo. Beds dip 5° SW. and lie near several normal faults.

J. Dark-brown to black shale probably from Mitten member of Pierre shale. Contains many small fragments of organic matter. SE. ¼ sec. 6, T. 44 N., R. 61 W., Weston County, Wyo. Beds dip 45° SW.

K. Gray mudstone about 100 feet below Monument Hill member of Pierre shale. Contains many small mollusks. Associated with a few calcareous concretions. NW. ¼ sec. 18, T. 44 N., R. 62 W., Weston County, Wyo. Beds dip 7° SW. and lie near plane of a normal fault.

Thin sections of these 11 samples and of 24 other specimens of shale and marl from the same beds in this region were cut, and the textural features, such as fissility and bedding laminations, were studied. The mineralogic examination included a study of these 35 thin sections, of a polished face of sample D, and of the crushed powders of these and about 10 other specimens.

MINERALOGY

The samples of shale, mudstone, marl, and limestone were examined both in thin section and with refractive-index liquids. The index liquids were used on crushed powders and on individual particles that had been disintegrated and sized in water.

SHALE, MUDSTONE, AND MARL

In every specimen examined, except those that were very calcareous, definitely crystalline micaceous clay is the chief constituent, and all the thin sections that were cut perpendicular to the bedding show a pro-

nounced aggregate orientation and positive elongation of these clay crystals parallel to the bedding. Many of these individual crystals do not lie exactly parallel to one another, but in all thin sections their average attitude is sensibly parallel to the bedding. Therefore, as the individual crystals show parallel extinction and as the slow ray vibrates in the plane of elongation, thin sections of the shale cut perpendicular to the bedding all show an aggregate positive elongation very much as they would if they were cut from single crystals.

The clay crystals are brown to gray and, having but one pronounced cleavage, are predominantly platy. Except for this cleavage, nearly all the crystals have rather indefinite outlines. Many grains seem to consist of groups of fibers lying in one plane; some crystals are slightly bent. The crystals range from less than 1 micron to as much as 100 microns in maximum diameter, and they are approximately equidimensional when lying upon their cleavage faces. In cross section, however, their length is commonly about seven times as great as their thickness.

Not all the clay crystals in any one sample have exactly the same refractive index, but the range is slight, and in all the samples studied the refractive index of the dominant clay mineral is between 1.55 and 1.57—in some slightly less, in others slightly more than 1.56. The maximum birefringence, as seen in thin sections cut perpendicular to the elongation, is about 0.02 or 0.03.

The dominant clay mineral seems to be the same in all the samples. Many characters, such as the parallel extinction, positive elongation, and crystal habit, are identical in all the samples examined, and the refractive indices and birefringences are relatively uniform. C. S. Ross, of the United States Geological Survey, identified this mineral as belonging to the beidellite³ type, or perhaps in the isomorphous series between beidellite and nontronite.⁴ He suggests that the rather high refractive indices may be due to a high content of Fe_2O_3 (indicated also by the brown color of the mineral), to adsorbed alkalis, or to loss of adsorbed water. Some of the small grains that show very definite crystal outlines may be anauxite.⁵

All the samples contain some and a few contain much fine quartz sand or silt. These quartz grains

are angular or subangular and range from 10 to 100 microns in diameter. Some of the samples contain a few flakes of muscovite and biotite.

All specimens from the Niobrara and Greenhorn formations and some specimens from the upper part of the Pierre shale contain noticeable amounts of relatively pure calcium carbonate ($\omega=1.66\pm$). It occurs in several forms—(a) as shells of globular foraminifers from 50 to 150 microns in diameter; (b) as spherulites from 4 to 7 and from 30 to 50 microns in diameter; (c) as minute lenses of microcrystalline calcite, 100 to 300 microns long, which lie parallel to the bedding; and (d) as sand grains and well-developed crystals from 30 to 100 microns in diameter. Specimens from the lower part of the Pierre shale contain spherulites of siderite from 5 to 50 microns in diameter.

Nearly all the thin sections examined are dark brown with organic matter, which consists chiefly of opaque dark-brown to black structureless fragments but also contains some transparent amber-colored masses of optically inactive material. These fragments and masses range in shape and size from equidimensional grains at the limit of visibility to very thin lenses more than 1 millimeter long that lie parallel to the bedding. A fragment of sample F, from which the mineral constituents were dissolved in hydrofluoric acid, also showed that the organic matter consists of particles of many different sizes.

A few very small nodules of finely crystalline pyrite were noted in most of the samples. Doubtless many other minor constituents are present in these rocks, but the small size of the particles and the lack of complete chemical analyses make their determination very difficult.⁶

PYRITIC LIMESTONE

Sample D, pyritic limestone from the Greenhorn formation, is sufficiently different in lithologic type to merit separate description. The chief constituents of this rock are calcium carbonate, pyrite, and gypsum. (See p. 11.) The calcium carbonate is present as large and small fragments of *Inoceramus* shells and as crystals of relatively pure calcite ($\omega=1.665\pm$) from 35 to 250 microns in diameter. The pyrite was determined with the reflecting microscope by C. S. Ross; it is minutely crystalline and occurs as isolated nodules, which range in size from the limit of visibility to several millimeters in diameter, and as surface coatings on some of the shell fragments. Some pyrrhotite or possibly ferrous sulphide is associated with the pyrite, for hydrogen sulphide is evolved when the sample is digested in dilute hydrochloric acid. Although usually a high-temperature mineral, pyrrho-

³ Beidellite, according to Mr. Ross, is the most widespread of the clay minerals.

⁴ Larsen, E. S., and Wherry, E. T., Beidellite, a new mineral name: Washington Acad. Sci. Jour., vol. 15, pp. 465-466, 1925. Ross, C. S., and Shannon, E. V., The chemical composition and optical properties of beidellite: Idem, pp. 467-468, 1925; The minerals of bentonite and related clays and their physical properties: Am. Ceramic Soc. Jour., vol. 9, pp. 93-96, 1926. Larsen, E. S., and Steiger, George, Dehydration and optical studies of alunogen, nontronite, and griffithite: Am. Jour. Sci., 5th ser., vol. 15, pp. 14-15, 1928.

⁵ Ross, C. S., and Foshag, W. F., Anauxite, A mineral species, based on material from Bilin, Czechoslovakia: Am. Mineralogist, vol. 13, pp. 153-155, 1928. Allen, V. T., Anauxite from the Ione formation of California: Idem, pp. 145-152.

⁶ See Grout, F. F., Relation of texture and composition of clays: Geol. Soc. America Bull., vol. 36, pp. 393-416, 1925.

tite not uncommonly occurs with other iron sulphides in fine-grained sedimentary rocks.⁷ and it can be formed in the laboratory with other iron sulphides at low temperatures.⁸ The gypsum in this limestone is microfibrinous and present chiefly in subparallel veinlets. (See pl. 5, *B*.) A lesser amount occurs as replacement films around shell fragments and calcite crystals in and immediately adjacent to pyrite masses. Some of the isolated crystals of calcite in these pyrite masses have been completely altered to gypsum. The pyrite masses also contain shells of Foraminifera, from 60 to 170 microns in diameter, that have been altered to gypsum and to iron oxide. Minor constituents of the rock are reddish iron oxide (bordering the pyrite masses), brownish bone phosphate (fragments of bones, teeth, and fish scales), and dark organic matter.

The gypsum in this sample is clearly secondary and, with the iron oxide, was very probably derived from the weathering of pyrite. The pyrite, on the other hand, was probably formed at the time of deposition of the rock or shortly thereafter. Its occurrence in isolated, minutely crystalline nodules and its intimate association with unpyritized shell fragments and perfect calcite crystals of different sizes (calcium carbonate being the substance it might most conceivably have replaced) strongly suggest that it formed on the sea floor or in the bottom oozes along with the fragments of shells and bones and the organic matter and carbonates. The partial corrosion indicated by the surface coatings of pyrite on some of the shell fragments probably took place soon after deposition in the waters or muds charged with hydrogen sulphide. (See p. 13.) The fact that the group of samples studied falls into a rough series in which the proportions of pyrite, organic matter, carbonates, and clay seem to be interrelated (see pp. 11-13) affords further evidence that the pyrite in this sample was formed during or soon after the deposition of the other constituents.

This sample came from the basal bed of the Greenhorn formation, and it contains an unusually large

amount of pyrite. Hence it accords with Goldman's observation⁹ that sulphides, like glauconite and phosphate, commonly occur at basal contacts. However, Goldman's hypothesis that these basal sulphides accumulate during periods of unusually slow sedimentation does not seem to account for this particular occurrence of pyrite. Detailed tracing of beds and many measurements of stratigraphic sections in the general vicinity of the outcrop from which sample D was collected show conclusively that this bed, like others in the Greenhorn formation, grades laterally within a few miles into calcareous concretions and black noncalcareous shale and that the calcareous beds are not measurably thinner than their shale equivalents,¹⁰ as the hypothesis would require. In fact, the available evidence indicates that the calcareous beds accumulated somewhat more rapidly than the shale beds. (See pp. 13, 52, 53.) It may be that waters which were escaping upward from the immediately underlying black shales as they were being compacted deposited some sulphide in the base of the calcareous muds and thus contributed to the large accumulation of pyrite in the basal bed of the limestone.

CHEMICAL COMPOSITION

ANALYSES

The percentage of organic matter in rocks that contain carbonate and hydrous minerals can not be determined by combustion nor by summing up total carbon, hydrogen, oxygen, and nitrogen. Methods specially adapted for differentiating between the organic and inorganic carbon and hydrogen in such rocks have been developed in the laboratories of the Bureau of Mines,¹¹ and fortunately it was possible to have the samples of Upper Cretaceous rocks from the Black Hills region analyzed in those laboratories.

⁷ Hatch, F. H., and Rastall, R. H., *The petrology of the sedimentary rocks*, pp. 201, 217, London, 1923.

⁸ Allen, E. T., Crenshaw, J. L., and Johnston, John, *The mineral sulphides of iron*: *Am. Jour. Sci.*, 4th ser., vol. 33, p. 214, 1912.

⁹ Goldman, M. I., *Lithologic subsurface correlation in the "Bend series" of north-central Texas*: U. S. Geol. Survey Prof. Paper 129, pp. 4-5, 1921; *Basal glauconite and phosphate beds*: *Science*, new ser., pp. 171-173, 1922; *Mississippian formations of San Saba County, Tex.*: U. S. Geol. Survey Prof. Paper 146, p. 56, 1926.

¹⁰ Rubey, W. W., *Cretaceous and Cenozoic formations on the north-west flank of the Black Hills*: U. S. Geol. Survey Prof. Paper — [in preparation].

¹¹ Fieldner, A. C., Selvig, W. A., and Taylor, G. G., *The determination of combustible matter in silicate and carbonate rocks*: *Bur. Mines Tech. Paper 212*, 1919.

Partial chemical analyses of Upper Cretaceous rocks from Black Hills region

[Analyst, H. M. Cooper, Bureau of Mines]

	A	B	C	D	E	F	G	H	I	J	K
Organic hydrogen-----	0.18	0.14	0.63	0.31	0.47	0.56	0.19	0.72	0.84	0.54	0.50
Organic carbon-----	1.20	1.40	2.12	2.29	.74	.81	1.47	3.08	1.14	5.07	1.75
Nitrogen-----	.09	.05	.11	.04	.11	.09	.11	.11	.11	.29	.14
Oxygen ^a -----	.94	.00	.00	.00	.00	.00	.93	2.51	.00	2.57	.00
Sulphur-----	.37	.28	.19	^b 16.71	.19	.13	.03	.89	.05	.48	.79
Carbon dioxide-----	.00	(^{c,d})	.00	19.98	4.32	.04	.11	27.46	2.41	.04	2.98
"Moisture"-----	4.95	2.46	2.60	3.54	2.49	3.42	2.34	1.00	1.90	4.78	3.92
"Combined" water-----	5.04	2.54	5.45	2.13	4.48	3.92	4.86	2.30	4.24	4.98	4.24
Ash-----	^e 87.23	93.37	89.57	^f 55.05	87.70	91.12	89.96	^g 61.93	^h 90.47	ⁱ 81.25	^j 86.08
	100.00	100.24	100.67	100.05	100.50	100.09	100.00	100.00	101.16	100.00	100.40
Chloroform-soluble-----	.06	.07	.08	.03	.05	.06	2.10	.18	.33	.20	.10
Calcium carbonate ^k -----	.00	^c .00	.00	45.41	9.82	.09	.25	62.42	ⁱ 6.35	.09	6.78
Organic matter ^l -----	2.41	1.59	2.86	2.64	1.32	1.46	2.70	6.42	2.09	8.47	2.39
Pyrite ^k -----	.69	.52	.36	^l 25.2	.36	.24	.06	1.66	.09	.90	1.48

^a Oxygen calculated by difference, probably a minimum value.

^b In part as SO₃.

^c CO₂ in another sample of Mowry shale=0.00 (U. S. Geol. Survey Prof. Paper 154, p. 157, 1929).

^d Not determined.

^e Ash minus the SO₃ found in the ash.

^f Ash minus the SO₃ and CO₂ found in the ash.

^g Contains oxidized iron from siderite.

^h Calcium carbonate calculated from CO₂.

ⁱ Ferrous carbonate instead of calcium carbonate.

^j Organic matter = H+C+N+O, probably a minimum value.

^k Pyrite calculated from S.

^l Allowing for S present as SO₃.

A. Black shale from Skull Creek member of Graneros shale.

B. Siliceous shale from Mowry member of Graneros shale.

C. Black shale from Belle Fourche member of Graneros shale.

D. Pyritic limestone from Greenhorn formation.

E. Calcareous marl from Greenhorn formation.

F. Gray shale from upper member (Turner sandy member) of Carlile shale.

G. Gray shale from lower member (Sage Breaks shale member) of Niobrara formation.

H. Calcareous marl from upper member (Beaver Creek chalky member) of Niobrara formation.

I. Ferruginous shale from lower part (Gammon ferruginous member) of Pierre shale.

J. Black shale from middle part (Mitten black shale member) of Pierre shale.

K. Gray shale from upper part of Pierre shale.

ORGANIC MATTER

Several of the hydrogen-carbon ratios are very high. Three of the samples (E, F, and I) show higher atomic ratios between these two elements than are shown by any known organic compounds, and re-determinations on these samples again gave high hydrogen contents.¹² This result suggests that inorganic hydrogen was not entirely eliminated by the method of analysis used.

The oxygen as given in these analyses was determined by difference, and as any oxidation of iron in pyrite, siderite, or clay minerals would increase the weight of the ash, the percentage of oxygen is therefore probably too low in every sample. Otherwise the low oxygen content of most of the samples would indicate that the organic matter consists largely of hydrocarbons instead of carbohydrate-like compounds,¹³ as is suggested by direct microscopic examination.

The total organic matter, taken as the sum of organic hydrogen, organic carbon, nitrogen, and oxygen in the different samples, ranges from 1.3 to 8.5 per cent by weight (about 2 to 14 per cent by volume).

¹² Selvig, W. A., personal communication.

¹³ For a table of the chemical compositions of organic compounds commonly found in sedimentary rocks see White, David, The carbonaceous sediments, in Twenhofel, W. H., Treatise on sedimentation, pp. 311-313, 1926.

As the oxygen percentages are probably too low, these totals are also probably minimum figures. It is of interest to note that the black shales, such as samples A and C, do not contain more than the average percentage of organic matter. Apparently the dark color is due quite as much to absence of carbonates as to abundance of organic matter.

The percentages of organic matter soluble in chloroform range from 0.03 to 2.10¹⁴ and average about 0.3 per cent. The maximum percentage corresponds to about 5 gallons of soluble bitumen to a ton of the rock. The samples appeared to be truly comparable, for no relation between these percentages and the time that had elapsed between collection and analysis could be detected.

It is noteworthy that these percentages of chloroform-soluble organic matter seem to vary stratigraphically; formations of Benton age, below the Niobrara formation (A to F), contain less and the higher formations (G to K) contain more than 0.09 per cent. Washburne¹⁵ and Geis¹⁶ have suggested that the Mowry shale may be an important source of light oils, but the single sample from this forma-

¹⁴ A check determination on sample G, made by E. T. Erickson, of the United States Geological Survey, gave 2.0 per cent.

¹⁵ Washburne, C. W., Some physical principles of the origin of petroleum: Am. Assoc. Petroleum Geologists Bull., vol. 3, pp. 357-359, 1919.

¹⁶ Geis, W. H., The origin of light oils in the Rocky Mountain region: Idem, vol. 7, pp. 499-504, 1923.

tion that was analyzed (B) yielded only 0.07 per cent by chloroform extraction. The apparent relation to stratigraphic position suggests that either increasing depth of burial or overburden does not increase but may even decrease the percentage of chloroform-soluble organic matter, or else the conditions of deposition, alteration after deposition, or the kind of organic matter itself changed progressively so as to obscure completely any increase due to increasing overburden.

The percentages of soluble organic matter seem to be related also to the degree of deformation that the rocks have undergone, as indicated in the following table:

Apparent relation of percentage of soluble organic matter to degree of deformation

Sample	Deformation		Percentage by weight of chloroform-soluble organic matter
	Dip of rocks where sampled	Proximity to faults	
G	50		2.10
J	45		.20
F	33		.06
H	10	(^a)	.18
Average			.64
K	7	(^a)	.10
D	6		.03
I	5	(^a)	.33
A	5		.06
Average			.13
C	4		.08
B	3		.07
E	1		.05
Average			.07

^a Near faults.

The exceptions to this average increase of chloroform-soluble matter with increase of dip may be caused by variations of other influencing factors, such as the stratigraphic position or depth, the amount and composition of the organic matter, and the extent to which the rock has yielded internally to the deformation to which it has been subjected. (See pp. 35, 38, 54.) Several of these factors seem to be operative, for not only is there a stratigraphic variation but the percentage of chloroform-soluble matter in a general way increases with the percentage of organic matter, and it also seems to vary with the composition of the organic matter.

This relationship, if it is a real and not merely an accidental one, suggests that the chloroform-soluble matter was generated in place or else made more soluble by the same processes that tilted the rocks. The more obvious alternative explanations, such as an inflow of liquid hydrocarbons into the rock or a de-

crease of inorganic constituents in the rock during folding, seem to be clearly inapplicable to the group of samples studied. Mead¹⁷ has suggested that, inasmuch as sandstones increase in volume when deformed, fluids may move into rocks that are being folded. However, as is discussed on pages 35 and 38, very fine-grained sediments such as shales probably decrease instead of increase in volume when deformed, and in the samples studied porosity seems to decrease with increasing dip of the rocks—that is, during folding fluids probably moved out of the rocks represented by these samples, not into them. The other alternative explanation mentioned above—that folding squeezes and hence decreases the percentage of inorganic constituents in a rock—could rest only upon a failure to distinguish between percentage by volume and percentage by weight. Although compacting during folding decreases the volume of a rock (pp. 35–38, 54), it might also decrease, and certainly would not increase, the percentage by weight of chloroform-soluble organic matter in the rock.

This suggestion that the chloroform-soluble matter may have been generated during folding is one that would be expected under the theory proposed by McCoy and Trager¹⁸ that the organic matter in a rock is progressively transformed into soluble bitumens by deformation. However, other geologists¹⁹ have reached different conclusions. McCoy's theory conforms with the evidence (pp. 35–36, 38, 54) that the rocks represented by these samples have been deformed internally by tilting and faulting.

If the percentage of soluble organic matter is an approximate measure of the oil in a rock and if the apparent relationship should prove to be a real and general one, it is of considerable economic importance, for it suggests that oil may be formed in any of the marine shales, mudstones, or marls in this region that have been sufficiently deformed by tilting or faulting. At first thought this suggestion seems to be contradicted by the results of drilling, for in this region oil has been found only in rocks of the Colorado group and older formations. However, a closer inspection indicates that this apparently contradictory evidence from drilling is by no means conclusive. For one thing, suitable reservoir rocks for the accumulation of petroleum are unknown in the Pierre shale throughout the southern part of the region, and they have not been penetrated by the drill where present farther

¹⁷ Mead, W. J., The geologic rôle of dilatancy: *Jour. Geology*, vol. 33, pp. 691, 697–698, 1925.

¹⁸ McCoy, A. W., Notes on the principles of oil accumulation: *Jour. Geology*, vol. 27, pp. 252–254, 1919. Trager, E. A., Kerogen and its relation to the origin of oil: *Am. Assoc. Petroleum Geologists Bull.*, vol. 8, pp. 301–311, 1924. McCoy, A. W., Soluble matter in oil shale: *Idem*, vol. 9, p. 1025, 1925.

¹⁹ Van Tuyl, F. M., and Blackburn, C. O., The effect of rock flowage on the kerogen of oil shale: *Idem*, vol. 9, pp. 158–164, 1925. Mead, W. J., and Hawley, J. E., The generation of oil in rocks by shearing pressures: *Am. Petroleum Inst. Bull.*, vol. 8, No. 54, p. 3, 1927.

north. Furthermore, in the four general districts in northeastern Wyoming (Newcastle, Osage, Thornton, and Moorcroft) where oil has been found in Upper Cretaceous rocks, its occurrence is closely related to minor faulting and steep dips.²⁰ Thus the results of drilling in themselves suggest that other districts of minor faulting and steep dips in the same region may be worth drilling.

The implications of this apparent increase of chloroform-soluble matter with increasing dip are so far-reaching as to indicate the desirability of examining it more critically and of determining if possible whether it is due to the causal relation assumed or to some more indirect or even fortuitous relation. As is discussed more fully on pages 35-38, the porosity as well as the content of chloroform-soluble matter in these rocks is related to their dip. In general the porosity decreases and the percentage of chloroform-soluble matter increases with increasing dip. Hence it might be expected that the percentage of chloroform-soluble matter would increase with decreasing porosity. Indeed, careful comparison shows a very rough relation of this sort, but it is not nearly as distinct as the relation of either porosity or chloroform-soluble matter to dip.

However, as the percentage of chloroform-soluble matter depends at least in part upon the percentage of total organic matter (p. 9), and as the percentage of total organic matter is different in the different samples, it may be a fairer test to use for comparison with the porosity the proportion of organic matter that is soluble in chloroform, instead of the gross percentage of chloroform-soluble matter. If this comparison is made, using the total carbon, hydrogen, and nitrogen (neglecting oxygen because of the uncertainty in its determination, p. 8) as an index of the amount of organic matter in the different samples, we find a rather distinct relationship.

Apparent relation between ratio of organic matter to chloroform-soluble matter and porosity

Sample	Total percentage by weight of organic carbon, hydrogen, and nitrogen	Percentage by weight of chloroform-soluble organic matter	Ratio of organic matter to chloroform-soluble matter	Porosity (percentage by volume)
C-----	2.86	0.08	35.8	33.3
J-----	5.90	.20	29.5	35.8
E-----	1.32	.05	26.4	37.6
A-----	1.47	.06	24.5	32.5
F-----	1.46	.06	24.3	23.8
K-----	2.39	.10	23.9	25.4
H-----	3.91	.18	21.7	25.4
I-----	2.09	.33	6.3	26.0
G-----	1.77	2.10	.8	25.3

²⁰ Rubey, W. W., The oil and gas possibilities of the Black Hills rim in Wyoming and Montana: U. S. Geol. Survey Bull. — [in preparation].

That is, the ratio of organic matter to chloroform-soluble matter seems to decrease with decreasing porosity. In other words, the more porous the rock the smaller the proportion of its organic matter that is soluble in chloroform. This apparent relation suggests that in the more porous samples a portion of the chloroform-soluble matter may have been lost by oxidation²¹ or carried away by percolating ground waters, an interpretation very different from the one based on the apparent relation between chloroform-soluble matter and dip.

The data available in this study do not warrant a decision as to which of these two possible explanations best accounts for the variations in the percentage of chloroform-soluble matter in these samples. The only conclusions that can safely be drawn are that the soluble organic matter in these rocks seems to increase with increasing dip and decreasing porosity. Inasmuch as the porosity appears to be fundamentally related to the dip, it is impossible to say whether the chloroform-soluble matter has been increased by deformation of the more highly tilted rocks or lost by oxidation or flushing from the more porous samples. The problem is of sufficient economic interest to justify further study.

PYRITE

The percentages of pyrite in the different samples may be estimated from the sulphur content. A small fraction of the sulphur may, of course, be combined with the organic matter. However, in nearly all the samples the ratio of sulphur to total organic matter is much too large for the sulphur to be accounted for in this way, and as disseminated pyrite was found in many of the samples by microscopic examination it seems that in this group of samples organic sulphur is probably negligible. Another portion of the total sulphur may be combined as gypsum, as in sample D; but in view of the fact that none was found in microscopic examination of the other samples and the probability that any such sulphates were formed by weathering of the pyrite (p. 7), it still seems justifiable to take the sulphur content as a measure of the pyrite once present in the unaltered rock. The presence of small quantities of either organic sulphur or sulphates would make the estimated percentage of pyrite too high. However, a third portion of the sulphur may be combined as pyrrhotite or as ferrous sulphide (see p. 6); and as this form of combination would make the estimated percentage of iron sulphide too low, the possible errors tend to offset one another. Computed thus, the pyrite content ranges from less than 0.1 to nearly 1.7 per cent in the shale and marl

²¹ Hawley, J. E., Generation of oil in rocks by shearing pressures: Am. Assoc. Petroleum Geologists Bull., vol. 13, pp. 313-314, 1929.

samples and to 25.2 per cent in the pyritic limestone (sample D).

CARBONATES

The chemical determinations of CO₂ and the microscopic determinations of the mineral species and purity of the carbonates afford the data necessary for computing the percentages of carbonate in the different samples. In all but one of the samples containing any carbonate it was found to be relatively pure calcite; in that one (sample I) the carbonate is siderite. The three samples from the Graneros shale contained no carbonate; the maximum was 62 per cent, in a sample of marl from the Niobrara formation. Next to the Niobrara and Greenhorn samples, those from the Pierre shale contain the most carbonate.

In considering these differences in the composition of the different formations, the possible importance of the large carbonate concretions in some of the shale members must not be overlooked. For example, the sample of shale from the lower or Sage Breaks member of the Niobrara formation (sample G) contains only 0.25 per cent of carbonate, but this member also contains many large concretions made up of relatively pure calcite, which were not represented in the sample. Similarly, the samples of shale from the Skull Creek and Belle Fourche members of the Graneros shale (samples A and C) contain no carbonate, but these shales are interbedded with zones of sideritic concretions. The lateral continuity or persistence of these and of other concretions in the region and the low carbonate content and relative impermeability²² of the inclosing shale beds lead the writer to believe with Tarr²³ that, for the most part, the concretions were formed before the muds were deeply buried. If this view is correct, the total carbonates in the entire member (concretions and all—not simply the carbonates in the shale) should be considered in comparing the different formations. However, rough estimates by the writer indicate that the total volume of the concretions is not sufficiently great to alter appreciably the order of relative carbonate contents that is indicated by the rock specimens alone.

COMPOSITION OF PYRITIC LIMESTONE

The CO₂ content indicates less than 50 per cent of carbonates in sample D, but in preparing it for mechanical analysis 73 per cent was found soluble in dilute hydrochloric acid. Microscopic examination indicated that the other soluble material consisted of

gypsum, bone phosphate, and iron oxide, and the evolution of H₂S in hydrochloric acid indicated some pyrrhotite. (See pp. 6-7.) With additional determinations, made by J. G. Fairchild, of the United States Geological Survey, of SO₃ (8.2 per cent) and P₂O₅ (less than 1 per cent), the probable composition of the rock was computed as follows:

	Per cent
Calcite.....	45.4
Pyrite.....	about 25.2
Gypsum.....	17.6
Iron oxide (computed as limonite).....	less than 6.1
Organic matter.....	more than 2.6
Bone phosphate.....	about 2.0
Moisture.....	1.1
	100.0

This composition, as computed from the analyses, seems fairly reliable, for it is independently checked within 1 per cent by both the acid-soluble and the ash determinations. It indicates that the ash, as corrected for SO₃ and CO₂, consists of two-thirds CaO, from calcite, gypsum, and bone phosphate, and one-third Fe₂O₃, from pyrite and limonite. It also indicates by the relative amounts of the different constituents that the gypsum was in part derived from the weathering of pyrite and calcite originally in the rock and in part introduced from outside. This conclusion seems to be borne out by the observation of both sulphatized carbonate grains and secondary veinlets of gypsum. (See pl. 5, *B*, and pp. 6-7.)

CONDITIONS OF DEPOSITION SUGGESTED BY CHEMICAL COMPOSITION

It is of especial interest to find that in a general way the percentages of carbonate vary with the percentages of organic matter and pyrite and that all three vary with stratigraphic position. In the following table the samples are arranged in order of increasing percentages of total carbonates and organic matter. This arrangement makes the samples fall into general stratigraphic groups, in which the average percentages of pyrite increase with the average percentages of carbonate and organic matter.

Percentages of total carbonates plus organic matter and of pyrite

Samples from the Colorado group, not including those from the calcareous parts of the Greenhorn and Niobrara formations

Sample	Carbonates plus organic matter	Pyrite
F.....	1.55	0.24
B.....	1.59	.52
A.....	2.41	.69
C.....	2.86	.36
G.....	2.95	.06
Average.....	2.3	.4

²²In fact, water was being squeezed out of the shale, rather than entering it, from the time of deposition until the time of uplift and erosion. (See pp. 35-38, 54.)

²³Tarr, W. A., Syngenetic origin of concretions in shale: *Geol. Soc. America Bull.*, vol. 32, pp. 373-384, 1921.

Percentages of total carbonates plus organic matter and of pyrite—Continued

Samples from the Pierre shale

Sample	Carbonates plus organic matter	Pyrite
I.....	8.44	0.09
J.....	8.56	.90
K.....	9.17	1.48
Average.....	8.7	.8

Samples from the calcareous parts of the Greenhorn and Niobrara formations

Sample	Carbonates plus organic matter	Pyrite
E.....	11.14	0.36
D.....	48.05	25.20
H.....	68.84	1.66
Average.....	42.7	9.1

The several interrelationships that bring about this grouping of the samples indicate that the conditions of deposition of the marine sediments in the Black Hills region during Upper Cretaceous time varied widely and that those conditions that were favorable for the preservation of carbonates were, in general, also favorable for the preservation of organic matter and pyrite.

This series into which all the samples seem to fall is probably suggestive of the range of conditions of deposition. In view of the fact that the carbonate consists partly of Foraminifera and molluscan shells, it is not surprising to find that the amount of organic matter varies with the amount of carbonate. Also the association of organic matter with iron sulphides is readily acceptable, for it has been noted commonly in recent sediments.²⁴ Even the association of carbonates and sulphides is not surprising, for there are theoretical reasons for believing that the stagnant waters favorable to the formation of iron sulphide may constitute one of the favorable environments for the accumulation of calcium carbonate. As demonstrated by Wells²⁵ and by Johnston and Williamson,²⁶

²⁴ Murray, John, and Renard, A. F., *Challenger Rept.*, Deep-sea deposits, p. 253, 1891. Murray, John, and Irvine, Robert, On the chemical changes which take place in the composition of the sea water associated with blue muds on the floor of the ocean: *Roy. Soc. Edinburgh Trans.*, vol. 37, p. 498, 1895. Harder, E. C., Iron-depositing bacteria and their geologic relations: *U. S. Geol. Survey Prof. Paper* 113, pp. 62-63, 1919. Goldman, M. I., "Black shale" formation in and about Chesapeake Bay: *Am. Assoc. Petroleum Geologists Bull.*, vol. 8, pp. 195-201, 1924. Bastin, E. S., The problem of the natural reduction of sulphates: *Am. Assoc. Petroleum Geologists Bull.*, vol. 10, pp. 1272-1280, 1926.

²⁵ Wells, R. C., The solubility of calcite in water in contact with the atmosphere and its variation with temperature: *Washington Acad. Sci. Jour.*, vol. 5, pp. 617-622, 1915; New determinations of carbon dioxide in water of the Gulf of Mexico: *U. S. Geol. Survey Prof. Paper* 120, pp. 1-16, 1919.

²⁶ Johnston, John, and Williamson, E. D., The rôle of inorganic agencies in the deposition of calcium carbonate: *Jour. Geology*, vol. 24, pp. 729-750, 1916.

For a discussion of the application of these principles to the interpretation of the conditions of sedimentation of shales see Rubey, W. W., Origin of the siliceous Mowry shale of the Black Hills region: *U. S. Geol. Survey Prof. Paper* 154, pp. 164-165, 1929.

the solubility of calcium carbonate depends largely upon the carbon dioxide content of sea water, which in turn depends chiefly upon the temperature of the water. The warmer (that is, in general, the shallower) the water, the less calcium carbonate it can dissolve. The surface water of the ocean has been found to be essentially saturated with respect to calcium carbonate. Hence it would seem that deeper waters would be unsaturated and that calcium carbonate could accumulate in deeper, colder water only where the rate of precipitation and burial exceeds the rate of solution or where the bottom waters are so stagnant that they become and remain locally saturated with respect to the carbonate. Otherwise circulation of the bottom waters would remove the saturated layer and would also bring dissolved oxygen into contact with organic matter, thus forming carbon dioxide and causing still more solution of the carbonate. This association of carbonate, organic matter, and pyrite therefore suggests the tentative hypothesis that the samples that contain the most of these constituents accumulated under anaerobic conditions.

However, this simple hypothesis does not meet all the facts satisfactorily. Foul, stagnant muds are not a favorable habitat for bottom-living organisms. The more highly calcareous and organic rock specimens contain the most fossils of bottom-living mollusks, and the less calcareous specimens are essentially barren of all fossils—not the reverse, as this hypothesis would indicate. Also anaerobic conditions of the sort postulated might be expected to yield deposits of siderite²⁷ instead of a mixture of calcium carbonate and iron sulphide. However, only one of the samples (I) contains detectable amounts of ferrous carbonate, and it is the less, not the more calcareous formations in this region that contain the more highly ferruginous carbonate concretions [Skull Creek shale (A), Belle Fourche shale (C), and Carlile shale (F)]. Furthermore, the preservation of very thin laminations suggests that the beds represented by the less calcareous samples (see pp. 51-52) were deposited in relatively quiet, not in relatively agitated (and therefore well-oxidized) water. Consequently, a somewhat different explanation for the association of calcium carbonate, organic matter, and pyrite must be sought.

A modified explanation is suggested by a separate consideration of the conditions favorable for preservation of each of these three constituents. The conditions theoretically most favorable for the preservation of calcium carbonate are warm waters (shallow or tropical), rapid burial, or stagnant saturated bottom waters. As the evidence seems to preclude the possibility of stagnant waters, there is at least a suggestion that the deposits represented by the more calcareous samples were formed at relatively shallow

²⁷ Harder, E. C., *op. cit.*, pp. 55, 72.

depths in warm, well-lighted, and well-oxidized water, where food supply and organisms were abundant and burial was rapid.

The amount of organic matter preserved in a sedimentary rock is the "product of an equation between the rate of supply of organic matter and the rate of decomposition";²⁸ it depends upon the chemical composition and the rate of growth of the organic matter, the rate of burial, and the rate of decay before and after burial. In general, relatively shallow water and moderately rapid sedimentation afford both a suitable habitat and optimum conditions for the preservation of organisms.

Ferrous sulphide is readily formed, in the presence of abundant organic matter and under reducing conditions, by the bacterial reduction of sulphates or by the action of hydrogen sulphide (chiefly from decaying protein) upon ferrous salts (bicarbonates, etc.) dissolved in sea water, upon ferric hydroxides previously deposited in the muds, or upon iron in clay minerals.²⁹ The ferrous sulphide is said to change over eventually into the more stable form, pyrite.

Combining these conclusions indicates that the conditions most favorable for the formation and preservation of calcium carbonate, organic matter, and pyrite together would be relatively shallow water and a rapid rate of accumulation and burial of organic matter. Under these conditions much organic matter and carbonate would be preserved by burial, and iron sulphide would form abundantly in the putrefying ooze—that is, chiefly below its upper surface.

This suggested explanation is more or less confirmed by studies of the present-day conditions of deposition of marine sediments. The blue muds are the predominant type of deposit in both deep and shallow water in all partly inclosed seas and for several hundred miles seaward from the oceanic coasts.³⁰

The materials of which the blue muds are principally composed are derived from the disintegration of continental land and are very complex in character. When collected this deposit is blue or slate-colored, with an upper red or brown colored layer, which had been in immediate contact with the water. The blue color is due to organic matter and sulphide of iron in a fine state of division, and these muds have, as a rule, when taken from the sounding tube or dredge, a smell of sulphureted hydrogen. The red or brown color of the thin watery upper layer is evidently due to the presence of ferric oxide or ferric hydrate, but as the deposit accumulates this

oxide is transformed into sulphide and ferrous oxide in the presence of organic matter in the underlying layers. * * * Sometimes the samples are homogeneous, at other times the aspect is heterogeneous, owing to the presence of large fragments of rocks and shells and small fragments of calcareous organisms. * * * They may contain from only a trace to 35 per cent of carbonate of lime.³¹

In general, the blue muds, like other oceanic deposits, show a gradual decrease in quantity of calcium carbonate with increase of depth.³² The amount of fine mud was found to range from 16 to 97 per cent.³³

The presence of sulphides and sulphureted hydrogen in all harbor muds, muddy bays near land, and, indeed, in nearly all the terrigenous deposits, such as the blue muds, is a sure indication that soluble and insoluble albuminoid and other organic matters are distributed throughout these muds and are in process of decomposition. Probably sulphides are present in all deep-sea deposits, but they are most abundant in muds near land, where there is rapid accumulation and where a large quantity of organic matter is borne down from the continents. In the red clays and the other truly pelagic deposits the quantity of organic matter is much less, and, owing to the slow accumulation, the sulphides are probably oxidized as soon as formed and never make up any considerable portion of the deposit.³⁴

Murray and Renard conclude that, of the different types of marine sediments, blue muds "not far removed from embouchures of large rivers" are accumulating the most rapidly.³⁵

From a study of the sea water associated with blue muds, collected from depths of 1 to 2, 3 to 5, and 16 fathoms, Murray and Irvine³⁶ concluded that calcium sulphate in the water is reduced to sulphides by bacterial decay of abundant organic matter and recombined with ferric oxide in the surface layer and with carbon dioxide from decaying organic matter so as to make the muds rich in iron sulphide and the sea water immediately above the muds more nearly saturated with respect to calcium carbonate. Similar conclusions were drawn by Van Delden³⁷ from an examination of black sulphide-bearing muds (overlain by a layer of light-colored mud a few millimeters thick) from canals in Holland.

Hence the association of calcium carbonate and pyrite in sediments is not inconsistent; in fact, the conditions favorable for the preservation of organic matter and sulphides also may be favorable for the growth and preservation of lime-secreting, bottom-living organisms. (See p. 53.)

²⁸ Goldman, M. I., op. cit., p. 200.

²⁹ Murray, John, and Irvine, Robert, On the chemical changes which take place in the composition of the sea water associated with blue muds on the floor of the ocean: Roy. Soc. Edinburgh Trans., vol. 37, pp. 481-507, 1895. Allen, E. T., Crenshaw, J. L., and Johnston, John, The mineral sulphides of iron: Am. Jour. Sci., 4th ser., vol. 33, pp. 169-236, 1912. Harder, E. C., op. cit., pp. 40-44, 60-64, 74-75, 82-84. Bastin, E. S., op. cit., pp. 1270-1299. Newhouse, W. H., Some forms of iron sulphide occurring in coal and other sedimentary rocks: Jour. Geology, vol. 35, pp. 73-83, 1927.

³⁰ Murray, John, and Renard, A. F., *Challenger* Rept., Deep-sea deposits, pp. 229-233, 1891.

³¹ Idem, p. 229.

³² Idem, p. 230.

³³ Idem, p. 231.

³⁴ Idem, p. 253.

³⁵ Idem, p. 411.

³⁶ Murray, John, and Irvine, Robert, On the chemical changes which take place in the composition of the sea water associated with blue muds on the floor of the ocean: Roy. Soc. Edinburgh Trans., vol. 37, pp. 481-507, 1895.

³⁷ Cited by Bastin, E. S., op. cit., p. 1275.

MECHANICAL ANALYSES

The mechanical composition, or the relative amounts of particles of the size of gravel, sand, and clay in a sedimentary rock, is one of its most significant lithologic characteristics. The proportions of grains of different sizes serve as an index not only of the general appearance and behavior of the rock under common conditions but also of its economic value and geologic history. This size distribution of particles is equally important in other fields of investigation, as in agriculture, ceramics, metallurgy, chemistry, and highway engineering and in the manufacture of cement, abrasives, paint, graphite, fertilizers, rubber, photographic emulsions, and of some drugs and foodstuffs. Consequently many methods for the determination of particle size are continually being proposed, and especially within the last 10 years much has been written on the subject. As many of the methods that geologists have used for determining the mechanical composition of fine-grained sedimentary rocks are in some respects unsatisfactory, a review of the methods that seemed applicable to such rocks is presented here to indicate the basis on which a method suitable for the particular study described in this paper was chosen.

REVIEW OF PROPOSED METHODS APPLICABLE TO FINE-GRAINED SEDIMENTARY ROCKS

The mechanical analysis of a sedimentary rock involves three more or less distinct problems: The rock must be prepared for study; the sizes of the particles must be measured; and the data must be presented in some readily usable form. In general, each of these problems is more difficult for the finer than for the coarser sediments.

PREPARATION OF SAMPLE

For studies in which microscopic examination and grain counting is a sufficiently accurate method of determining the mechanical composition, hand specimens of the rock may be examined under the lens directly, thin sections may be cut,³⁸ or samples may be disintegrated for a more thorough study of the individual grains. But for the examination of many common types of sedimentary rocks, some more objective and mechanical method of analysis is probably more accurate. The essential preparation for these more objective analyses is a thorough disintegration of the rock—that is, a thorough separation of the individual rock particles. Some rocks disintegrate

readily in water or even in air. Others, such as siliceous shales and silicified sandstones, can not be disintegrated without methods that so change the size and number of the grains that mechanical analyses are then worthless. Most fine-grained sedimentary rocks require some sort of special treatment to separate the individual particles.

The method of disintegration to be used depends largely upon the object of the analysis. If the present composition of the rock is desired for descriptive or mineralogic purposes, all secondary minerals should be retained. If, however, the mechanical composition at the time of deposition is sought, these secondary minerals must be excluded. For many geologic investigations the goal is this original composition, because from it inferences can be drawn as to the velocity of currents and other conditions at the time of sedimentation. Of course it is not always possible to determine just which minerals are secondary, and even if they are known it is commonly difficult to remove them without greatly altering the original constituents. For example, if the rock examined is a sandstone cemented with calcite, the investigator must know that the sand grains are not carbonates before he proceeds to remove the cement with acid. In clay rocks the difficulties are even greater. At the time of sedimentation most of the clay particles may have been flocculated by salts dissolved in the water³⁹ into aggregates which settled as units, and thus the present size of the individual particles would give no clue to the size of the original aggregates. Also the clay particles may have been very small at the time of deposition, but after burial they may have grown into much larger crystals of clay minerals.⁴⁰ Thus the present size of the clay particles may differ greatly from their original size. Furthermore, even the present composition is difficult to determine, as it is nearly impossible to disintegrate most clay rocks completely. Crushing may fracture the delicate platy crystals, heating may break down the hydrous clay compounds, and chemicals or even pure water may dissolve or cause chemical changes in the finer particles.

In view of these uncertainties and difficulties, it is clearly impossible to attain anything more than an approximation to the initial size distribution of particles in clay rocks. Some methods of preparation, measurement, and presentation give more accurate results than others, but it is useless to seek methods that are much more precise than the least accurate or most uncertain step in the entire determination.

³⁸ For methods particularly adapted to cutting thin sections of shale and clay, see Sayles, R. W., Microscopic sections of till and stratified clay: *Geol. Soc. America Bull.*, vol. 32, pp. 59-62, 1921; Ross, C. S., A method of preparing thin sections of friable rock: *Am. Jour. Sci.*, 5th ser., vol. 7, pp. 483-485, 1924; Methods of preparation of sedimentary materials for study: *Econ. Geology*, vol. 21, pp. 460-468, 1926.

³⁹ Grabau, A. W., Principles of stratigraphy, pp. 655-657, 1913.

⁴⁰ Leith, C. K., and Mead, W. J., Metamorphic geology, pp. 107-108, 1915. Lewis, J. V., Fissility of shale and its relation to petroleum: *Geol. Soc. America Bull.*, vol. 35, p. 578, 1924. Twenhofel, W. H., Treatise on sedimentation, p. 186, 1926.

For most of the clay rocks the uncertainty about the degree of flocculation during sedimentation and the amount of secondary growth of clay particles since deposition is probably greater than the errors in some of the methods of disintegration; likewise the unavoidable errors in even the best methods of disintegration probably exceed the errors in most of the methods of measurement and presentation of results.

Many investigations have been made, especially by soil scientists, of the methods of disintegrating soils and clays, but no simple expeditious procedure that gives satisfactory results seems yet to have been found. Nearly all fine-grained rocks and soils require chemical treatment to loosen the cement or deflocculate the grains. Soil physicists recommend that carbonates and humus, which may cement the finer particles, should be removed with dilute hydrochloric acid and hydrogen peroxide or nitric acid.⁴¹ However, geologists will probably agree that, as the carbonates and organic matter may be essential constituents of the rock, they should, if possible, be retained in the mechanical analysis. Furthermore, in the treatment with hydrochloric acid some of the finest clay material is dissolved.⁴² Most geologists, soil physicists, and other investigators have agreed that the tendency of small clay particles to gather together in floccules⁴³ must be overcome by washing out the electrolytes in the sample with distilled water⁴⁴ or by neutralizing the water with some chemical, usually an alkali (ammonia, ammonium hydroxide, sodium carbonate, sodium hydroxide, potassium hydroxide, barium hydroxide, water glass, soap, or pyrogallol⁴⁵). Washing out the electrolytes is a tedious process, and if it is attempted great care must be taken to avoid loss of the finer clay particles. Of the chemicals that may be added for neutralization, ammonia and sodium carbonate seem most effective, most easily handled, and

least harmful to the sample. Sodium carbonate is highly effective⁴⁶ but dissolves more fine particles of silica than ammonia⁴⁷ does. Ammonia is said to cause rather than prevent flocculation, if calcium or magnesium carbonate is present in large quantities.⁴⁸ Concentrations ranging from 0.007 to 2.5 per cent of ammonia have been used by different investigators; the lower concentrations give most complete dispersions, but somewhat higher concentrations may be desirable, especially if organic matter and carbonates are present, in order to offset loss by evaporation and to maintain alkalinity throughout the analysis.⁴⁹ Puri and Keen⁵⁰ obtained most nearly complete dispersions in suspensions of 1 part soil to 100 or more parts of water.

In addition to the chemical treatment, most fine-grained rocks or soils must be subjected to mechanical agitation to separate the individual grains. This agitation may be accomplished by shaking in the hand; by some mechanical shaking, vibrating,⁵¹ or stirring⁵² device, or by boiling. Samples that do not yield to this treatment must be rubbed gently with the finger, a rubber pestle, or a brush. Violent methods such as crushing with mortar and pestle or heating to redness and disrupting by sudden immersion in cold water almost certainly defeat the purpose of the mechanical analysis. Experiments indicate that, as might be expected, differences in mechanical treatment produce widely different results in deflocculation and that the rubber-pestle and brush methods, though liable to subjective errors, are probably most effective.⁵³

⁴¹ Atterberg, A., Die rationelle Klassifikation der Sande und Kiese; Chem. Zeitung, Jahrgang 29, p. 195, 1905. Hissink, D. J., Methods of mechanical analysis of soils: Internat. Soc. Soil Sci. Proc., vol. 1, pp. 705-724, 1925. Report of the mechanical analysis subcommittee of the Agricultural Education Association—The mechanical analysis of soils; a report on the present position and recommendation for a new official method: Jour. Agr. Sci., vol. 16, pp. 123-144, 1926.

⁴² Hissink, D. J., op. cit., pp. 712-713. Novák, V., and Smolík, L., Sur la quantité et la composition chimique de l'argile colloïdale des sols: IV^{ème} Conférence Internat. pédologie [Rome] Actes, vol. 2, pp. 128-141, 1926.

⁴³ Wentworth, C. K., Methods of mechanical analysis of sediments: Iowa Univ. Studies, vol. 11, pp. 42-43, 1926.

⁴⁴ Wiegner, G., Ueber den Einfluss verschiedener Vorbehandlungsmethoden auf den mit Hilfe des Schlämmapparates von Wiegner-Gessner ermittelten Dispersitätsgrad von Bodensuspensionen: IV^{ème} Conférence Internat. pédologie [Rome] Actes, vol. 2, pp. 87-102, 1926.

⁴⁵ Briggs, L. J., Martin, F. O., and Pearce, J. R., The centrifugal method of mechanical analysis: U. S. Dept. Agr. Bur. Soils Bull. 24, pp. 22-25, 1904. Atterberg, A., op. cit. Boswell, P. G. H., The separation of the finer constituents of sedimentary rocks: Faraday Soc. Trans., vol. 18, p. 38, 1922. Holmes, A., Petrographic methods and calculations, pt. 1, p. 186, 1923. Sayles, R. W., Seasonal deposition in marine waters: Nat. Research Council Comm. on Sedimentation Rept. [Apr. 18, 1923], p. 63, 1923. Biehlinger, A. V., The properties of clays: Second Colloid Symposium Mon., p. 88, Chemical Catalog Co., 1925.

⁴⁶ Puri, A. N., and Keen, B. A., Dispersions of soil in water under various conditions: Jour. Agr. Sci., vol. 15, pp. 157-158, 1925.

⁴⁷ Steiger, George, in Twenhofel, W. H., and others, Treatise on sedimentation, p. 632, 1926.

⁴⁸ Briggs, L. J., Martin, F. O., and Pearce, J. R., op. cit., p. 24.

⁴⁹ Fletcher, C. C., and Bryan, H., Modification of the method of mechanical soil analysis: U. S. Dept. Agr. Bull. 84, p. 9, 1912.

⁵⁰ Puri, A. N., and Keen, B. A., op. cit., p. 153.

⁵¹ Whittles, C. L., The determination of the number of bacteria in soil; II, Methods for the disintegration of soil aggregates and the preparation of soil suspensions: Jour. Agr. Sci., vol. 14, pp. 346-369, 1924.

⁵² Bonyoucos, G. J., Directions for determining the colloidal material of soils by the hydrometer method: Science, new ser., vol. 66, pp. 16-17, 1927.

⁵³ Beam, W., The mechanical analysis of arid soils: Cairo Sci. Jour., vol. 5, p. 107, 1911. Atterberg, A., Die mechanische Bodenanalyse und die Klassifikation der Mineralböden Schwedens: Internat. Mitt. Bodenkunde, Band 2, p. 314, 1912. Riecher, G., Die Ausführung mechanischer und physikalischer Bodenanalysen: Idem, Band 6, pp. 193-208, 318-346, 1916. Koettgen, P., Zur Methodik der physikalischen Bodenanalyse: Idem, Band 7, pp. 205-246, 1917. Nolte, O., Der Einfluss des Kochens und des Schüttelns auf feine Mineralteilchen—ein Beitrag zur Ausführung von mechanischen Bodenanalysen: Landw. Vers. Stat., Band 93, p. 247, 1919. Odén, Sven, Bodenkundliche Forschungen an dem chemischen Universitäts-Laboratorium der Universität Upsala, 1914-1919—III, Ueber die Vorbehandlung der Bodenproben zur mechanischen Analyse: Internat. Mitt. Bodenkunde, Band 9, pp. 301-418, 1920. Hissink, D. J., Die Methode der mechanischen Bodenanalyse: Idem, Band 11, pp. 1-11, 1921. König, J., and Hasenbäumer, J., Zur Beurteilung neuer Verfahren für die Untersuchung des Bodens: Landw. Jahrb., Band 56, p. 439, 1921. Gile, P. L., Middleton, H. E., Robinson, W. O., Fry, W. H., and Anderson, M. S., Estimation of colloidal material in soils by adsorption: U. S. Dept. Agr. Bull. 1193, pp. 16-17, 1924. Puri, A. N., and Keen, B. A., op. cit., p. 159.

In nearly all these methods disintegration is sought within a few days after the clay or soil is put into water. Some facts indicate that disintegration is attained more easily by a longer period of soaking. It seems that the readiness with which a soil or clay disintegrates depends largely upon the moisture content of the sample. Air-dried samples are much harder to disintegrate than samples examined in the natural moist condition.⁵⁴ Dispersability, especially in clays, decreases with decreasing water content or with heating above the air-dried condition, and rewetting does not seem to make the dispersion easier.⁵⁵ Hilgard⁵⁶ noted that Recent sediments disintegrate much more easily than ancient sediments. Whittles⁵⁷ found it advisable to moisten soil samples slowly in order to avoid lumpiness.

These observations, like those of Terzaghi⁵⁸ and Hedberg⁵⁹ on the compacting of muds and sands, indicate that the induration of clay is due largely to loss of moisture or of pore space—that is, to compacting by drying or by pressure—and that a considerable period of time is required for a clay rock to take up again the moisture it is capable of holding at some higher humidity or lower pressure. The inference therefore seems justified that a long period of soaking in water assists in the disintegration of clay rock. This conclusion is borne out by the writer's experience in disintegrating samples of Cretaceous shale and bentonite.⁶⁰ However, a long period of soaking increases the likelihood of chemical solution of the smallest particles and may cause some hydration of the minerals.

METHODS OF MEASUREMENT

Mechanical analyses of granular mixtures, as generally understood, give the percentages of different-sized particles in a sample. In natural mixtures, such as sedimentary rocks, particles of all sizes between the largest and smallest are usually present. By this generally accepted definition, a statement of the average size of all the particles in such a rock would not be a mechanical analysis. The percentage of grains larger and smaller than some arbitrary intermediate limit might be so considered, however; and as the

number of these arbitrary limits is increased the analysis approaches a complete statement of the size distribution within the sample. Obviously the more nearly complete the analysis the greater its usefulness, for thus the degree of uniformity of the sizing and any irregularities in the size distribution become more apparent. On the other hand, the more nearly complete the analysis the more laborious the determination. Fortunately, some methods give a moderately complete analysis with one manipulation, whereas others require repeated separations or a multiplication of apparatus to attain the same degree of refinement.

Aside from those methods that determine a single physical property of a granular substance, such as the average diameter⁶¹ or the total internal surface of the particles,⁶² and those methods that are especially adapted for submicroscopic particles, such as those that measure the amplitude or effectiveness of the Brownian movement,⁶³ there are three principal methods for the mechanical analysis of mixtures containing small particles—separation by sieves, microscopic counts, and separation based on the different settling velocities of large and small particles in a liquid. Each method measures slightly different properties of particles and is best suited for certain sizes, but it is well to check one method against the others to avoid serious errors in interpretation.

SIEVES

The size of an equidimensional hole through which a particle will pass is determined not by the maximum or minimum dimension of the particle, but chiefly by the intermediate dimension. If a mixture of particles is satisfactorily disintegrated and the sizes of the holes in several sieves are known, the proportions of grains whose intermediate dimensions are greater and less than each sieve size can be determined with relative ease and sufficient accuracy.⁶⁴ However, the method is not applicable to very small particles, for Holmes states that the smallest grains that can satisfactorily

⁵⁴ Ehrenberg, P., and Van Zyl, J. P., Weitere Untersuchungen über die Beschaffenheit der Bodenkrümmel: Internat. Mitt. Bodenkunde, Band 7, pp. 90–103, 1917; Band 8, pp. 41–49, 1918.

⁵⁵ Puri, A. N., and Keen, B. N., op. cit., pp. 150–151, 153, 156.

⁵⁶ Hilgard, E. W., On the silt analysis of soils and clays: Am. Jour. Sci., 3d ser., vol. 6, p. 335, 1873.

⁵⁷ Whittles, C. L., op. cit., p. 366.

⁵⁸ Terzaghi, Charles, Principles of soil mechanics: Eng. News-Record, vol. 95, pp. 742–746, 796–800, 832–836, 874–878, 912–915, 987–990, 1026–1029, 1064–1068, 1925.

⁵⁹ Hedberg, H. D., The effect of gravitational compaction on the structure of sedimentary rocks: Am. Assoc. Petroleum Geologists Bull., vol. 10, pp. 1035–1072, 1926.

⁶⁰ Rubey, W. W., Cretaceous and Cenozoic formations on the north-west flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [in preparation].

⁶¹ Anderson, M. S., and Mattson, Sante, Properties of colloidal soil material: U. S. Dept. Agr. Bull. 1452, p. 4, 1926. Stutz, G. F. A., and Pfund, A. H., A relative method for determining particle size of pigments: Ind. and Eng. Chemistry, vol. 19, pp. 51–53, 1927. Clark, G. L., X-rays and colloids: Colloid Symposium Mon., vol. 4, pp. 156–162, 1926.

⁶² Sauramo, Matti, Studies on the Quaternary varve sediments in southern Finland: Comm. géol. Finlande Bull. 60, pp. 17–19, 1923. Zunker, F., Die Bedeutung und Bestimmung der spezifischen Oberfläche des Bodens: IV^{ème} Conférence internat. pédologie [Rome] Actes, vol. 2, pp. 238–249, 1926.

⁶³ Wightman, E. P., and Sheppard, S. E., The size-frequency distribution of particles of silver halide in photographic emulsions and its relation to sensitometric characteristics—II, The methods of determining size-frequency distribution: Jour. Phys. Chemistry, vol. 25, pp. 562, 569, 1921. Burton, E. F., and Reid, B. M., Determination of the size of colloidal particles by means of alternating electric fields: Philos. Mag., vol. 50, pp. 1221–1226, 1925.

⁶⁴ Wentworth, C. K., The accuracy of mechanical analyses: Am. Jour. Sci., 5th ser., vol. 13, pp. 399–408, 1927.

be separated with sieves are about 0.25 millimeter in diameter⁶⁵ ("fine sand" in most systems of classification). By using the most carefully made sieves and by long-continued shaking or by sieving under water to escape the aggregating effect of hygroscopic moisture and electrical charges, particles as small as 0.05 millimeter in diameter ("very fine sand" or "coarse silt" in most systems of classification) can perhaps be separated by this method.⁶⁶ But, as most of the particles in the common fine-grained sedimentary rocks are still smaller than this lower limit, some method other than sieving must be used for their mechanical analysis.

The principle of sieving is at least theoretically applicable to much smaller sizes by the use of filters, for the pores in ordinary and hardened filter paper approximate the dimensions⁶⁷ of the limiting diameter between silt and clay. However, little or no work seems to have been done on this possible method.

MICROSCOPIC COUNTS

The method of direct microscopic counts or estimates of the proportions of different-sized grains recommends itself to most geologists because it is free from many of the uncertainties of the more indirect methods of measurement and because it involves little work other than that necessary in routine petrographic examinations. These advantages are especially great now that a method for cutting satisfactory thin sections of soft, fine-grained rocks has been developed.⁶⁸ In certain other respects, however, microscopic counts are less satisfactory. First, the method yields only approximate results unless great pains and much time are taken in making the counts. Second, it is very difficult to resolve aggregates of silt and clay-sized particles into their constituent particles, even under high magnifications. Third, errors of sampling are much greater in a very thin fragment of the rock than in a larger specimen—that is, there is much greater likelihood of obtaining a representative sample of the rock in a 10-gram fragment than in a thin section, as one is several thousand times as large as the other. Finally, it is questionable whether or not microscopic methods measure features of as great geologic significance as some of the indirect methods. If, as seems certain, most of the grains in an argillaceous rock are irregularly shaped, the processes of sedimentation and compaction have probably caused them to lie with their flatter sides parallel to the bedding. Thus thin sections cut parallel to the bedding show the two larger dimensions, and sections cut across the bedding the minimum dimension. For

most geologic studies the combined effect of all three dimensions and of the surface area and density upon the settling velocity in water or in air is of greater importance than the diameter in some one plane. Nevertheless, microscopic examinations are essential as a means of verifying and calibrating the indirect methods, and for those rocks that can not be satisfactorily disintegrated microscopic examination is the only method for determining grain size.

Udden⁶⁹ recommended counting the relative numbers of grains in each size group smaller than 0.125 millimeter in diameter, calculating their weight as if they were perfect spheres, and reducing to 100 per cent. The Rosiwal method⁷⁰ of counting those grains that fall on some random straight line is familiar to most geologists. Wentworth⁷¹ suggests projecting the microscopic image onto a ground-glass surface for greater ease in counting. Photomicrographs or camera-lucida drawings may be measured with the planimeter⁷² or by direct measurement when projected upon a screen.⁷³ The average size of particles in different fractions (previously separated by some other method) may be determined with blood-count chambers,⁷⁴ gelatin mounts,⁷⁵ a checker-work eyepiece micrometer,⁷⁶ or the ultramicroscope.⁷⁷

SETTLING VELOCITIES

GENERAL FEATURES OF THE PROBLEM

A grain of sand, silt, or clay falling through water has for a moment an accelerating velocity, but, the resistance of the water quickly balancing the acceleration, the velocity becomes uniform as the grain continues its fall. Large grains, of course, fall faster than small grains and attain much greater uniform velocity. The Stokes law states that among spherical grains of equal density this uniform velocity varies as the square of the diameter. This law agrees fairly

⁶⁵ Udden, J. A., Mechanical composition of elastic sediments: *Geol. Soc. America Bull.*, vol. 25, pp. 658-659, 1914.

⁷⁰ Lincoln, F. C., and Rietz, H. L., The determination of the relative volumes of the components of rocks by mensuration methods: *Econ. Geology*, vol. 8, pp. 120-139, 1913. Johannsen, A., and Stephenson, E. A., On the accuracy of the Rosiwal method for the determination of the minerals in a rock: *Jour. Geology*, vol. 27, pp. 212-220, 1919. Wentworth, C. K., An improved recording micrometer for rock analysis: *Idem*, vol. 31, pp. 228-232, 1923. Jeffries, Z., Kline, A. H., and Zimmer, E. B., The determination of grain size in metals: *Am. Inst. Min. Eng. Trans.*, vol. 54, p. 604, 1916.

⁷¹ Wentworth, C. K., Methods of mechanical analysis of sediments: *Iowa Univ. Studies*, vol. 11, p. 38, 1926.

⁷² Johannsen, A., A planimeter method for the determination of the percentage composition of rocks: *Jour. Geology*, vol. 27, pp. 276-285, 1919.

⁷³ Green, Henry, A photographic method for the determination of particle size of paint and rubber pigments: *Franklin Inst. Jour.*, vol. 192, pp. 637-666, 1921.

⁷⁴ Wightman, E. P., and Sheppard, S. E., *op. cit.*, pp. 571-586.

⁷⁵ Renwick, F. F., and Sease, V. B., An improved method of sedimentary analysis applied to photographic emulsions: *Second Colloid Symposium Mon.*, pp. 37-45, Chemical Catalog Co., 1925.

⁷⁶ Fry, W. H., The microscope estimation of colloids in soil separates: *Jour. Agr. Research*, vol. 24, pp. 879-883, 1923.

⁷⁷ Svedberg, T., and Nichols, J. B., Determination of size and distribution of size of particles by centrifugal methods: *Am. Chem. Soc. Jour.*, vol. 45, pp. 2010-2017, 1923.

⁶⁶ Holmes, A., *Petrographic methods and calculations*, pt. 1, p. 201, 1923.

⁶⁷ Nutting, P. G., and Wentworth, C. K., personal communications.

⁶⁸ Ayres, E. E. jr., Subsidence in colloidal systems, in Alexander, Jerome, *Colloid chemistry*, vol. 1, p. 855, Chemical Catalog Co., 1926.

⁶⁹ Sayles, R. W., *op. cit.* Ross, C. S., *op. cit.*

well with experimental results on approximately spherical grains that have densities near that of quartz and diameters greater than 0.0002 millimeter⁷⁸ and less than about 0.085 millimeter⁷⁹ or 0.2 millimeter.⁸⁰ Grains with diameters greater than about 1.5 millimeters follow another rule and fall with velocities proportional to the square root of the diameter.⁸¹ Grains of intermediate sizes, with diameters between these two limits (about 0.2 and about 1.5 millimeters—that is, essentially “sand” grains), fall with velocities that seem to follow no simple law. Although the relation between settling velocities and diameters of grains of all sizes and different shapes can not yet be stated precisely, the very marked effect upon settling velocities of slight differences in the size of small particles affords the basis for a group of methods of sizing granular mixtures. Because of the fundamental principle involved, settling in water, these methods are especially significant in studies of sedimentary rocks.

Settling velocities depend not only upon the diameter of the grains but also upon the viscosity and density of the suspending fluid, and therefore grains that fall too rapidly in water for accurate measurement or calibration may be measured in more viscous⁸² or denser liquids. Similarly, particles that fall too slowly for convenient measurement may be speeded up by using a less dense or less viscous fluid or even air,⁸³ or else the effect of gravitation may be increased by settling within a high-speed centrifuge.⁸⁴

The number of particles in the suspension (the concentration) is another but related factor in the settling velocity of a particle.⁸⁵ High concentration causes interference between particles, flocculation, and vertical currents, and it appreciably alters the viscosity and density of the fluid, so that in very muddy waters the rate of fall is retarded. However, experiments indicate that this factor is negligible for concentrations of less than 1 or 2 per cent.⁸⁶

⁷⁸ Perrin, M. J., *Brownian movement and molecular reality*, pp. 34–40, London, 1910.

⁷⁹ Allen, H. S., *Motion of a sphere in a viscous fluid*: *Philos. Mag.*, 5th ser., vol. 50, pp. 323–338, 1900.

⁸⁰ Richards, R. H., *Ore dressing*, vol. 3, p. 1425, 1909.

⁸¹ Rittinger, P. R. von, *Aufbereitungskunde*, pp. 165, 325, 1867; cited by Richards, R. H., *op. cit.*, vol. 1, pp. 472, 476.

⁸² Wightman, E. P., and Sheppard, S. E., *op. cit.*, pp. 571–586.

⁸³ Whympere, R., *Analytical elutriation methods of separating cacao husk from cocoa powder*: *Faraday Soc. Trans.*, vol. 18, p. 49, 1922.

⁸⁴ Briggs, L. J., Martin, F. O., Pearce, J. R., *The centrifugal method of mechanical soil analysis*: U. S. Dept. Agr. Bur. Soils Bull. 24, 1904. Svedberg, T., and Nichols, J. B., *Determination of size and distribution of size of particles by centrifugal methods*: *Am. Chem. Soc. Jour.*, vol. 45, pp. 2910–2917, 1923.

⁸⁵ Barus, Carl, *Subsidence of fine solid particles in liquids*: U. S. Geol. Survey Bull. 36, pp. 19–20, 1886. Richards, R. H., *op. cit.*, pp. 610, 626–627. Robinson, C. S., *Some factors influencing sedimentation*: *Ind. and Eng. Chemistry*, vol. 18, pp. 869–871, 1926.

⁸⁶ Schloesing, T., sr., *Sur l'analyse mécanique des sols*: *Compt. Rend.*, vol. 136, pp. 1603–1613, 1903. Odén, Sven, *On clays as disperse systems*: *Faraday Soc. Trans.*, vol. 17, p. 334, 1922. Robinson, G. W., *A new method for the mechanical analysis of soils and other dispersions*: *Jour. Agr. Sci.*, vol. 12, pp. 306–321, 1922.

Theoretically the simple way to determine particle sizes from the different settling velocities would be to start a mixture of different-sized particles falling from the top of a fluid simultaneously, and then by some device to measure at different times the amounts that had fallen to a certain depth. Practically, however, it is almost impossible to introduce all the particles simultaneously at the top of the fluid. Slight differences in depth of particles at the time of introduction cause considerable errors in the result, if the distance between the top and bottom of the fluid is small. If the fluid is made deeper, so as to reduce the effect of these differences in original depth, particles do not accumulate on the bottom in measurable quantities for several days, and during this period changes of temperature greatly alter the viscosity of the fluid and set up convection currents that materially affect the settling velocities of the smaller particles. (See pp. 20, 28.) Also the likelihood of contamination with dust increases with a longer settling time, and the experimental work is much less convenient because spread over a longer time.

The means by which this difficulty is met may be taken as a basis for classifying the different settling methods. (a) In the elutriation methods a rising current of known velocity passes through a suspension of particles and carries away those particles whose settling velocity is less than the current, regardless of their original depth in the fluid. The remaining particles are then dried and weighed. (b) In the decantation methods no effort is made to introduce the particles at some one height, but the mixture is repeatedly stirred, settled, and decanted, thus eventually eliminating nearly all particles whose settling velocity is less than some chosen rate.⁸⁷ Here also the remaining particles are dried and weighed. (c) In the so-called “sedimentation” methods particles are not washed away by a rising current or by decantation, but the rate at which the sediment accumulates at the bottom or falls out of a certain part of the suspension is noted at frequent intervals. Two totally different methods for introducing the particles into the fluid are employed. Either the sample is mixed with a small amount of fluid and this very concentrated suspension is introduced carefully into the top of the settling fluid, in an endeavor to start all particles settling from some one depth simultaneously;⁸⁸ or else the sample is thoroughly mixed with all the fluid in an endeavor to attain a uniform distribution

⁸⁷ Wentworth, C. K., *Methods of mechanical analysis of sediments*: *Iowa Univ. Studies*, vol. 11, pp. 41–42, 1926.

⁸⁸ Audubert, R., and Rabaté, H., *Sur une méthode de détermination de la répartition granulométrique des systèmes dispersés*: *Compt. Rend.*, vol. 180, pp. 1663–1665, 1925. Werner, Donovan, *A simple method of obtaining the size distribution of particles in soils and precipitates*: *Faraday Soc. Trans.*, vol. 21, pp. 388–389, 1926. Calbeck, J. H., and Harner, H. R., *Particle size and distribution by sedimentation methods*: *Ind. and Eng. Chemistry*, vol. 19, pp. 58–61, 1927.

of particles throughout the suspension. In the latter method, as also in the decantation methods, the total sediment which in any given time has fallen to a certain depth consists of large particles which have completely settled out of the suspension and of a certain amount of smaller particles which started at intermediate depths and had not so far to fall. The amount of these smaller particles can be calculated mathematically; or, more conveniently, the true composition of the sample can be determined quickly, simply, and accurately by graphic means, as is set forth more fully on pages 24-25.

ELUTRIATION METHODS

The elutriation methods have long been used for analyzing soils and clays.⁸⁹ In one modification the grains are not dried and weighed, but their volume is read on a graduated scale at the bottom of the vessel.⁹⁰ In another modification the elutriation is accomplished through siphons of different sizes hanging in the suspension.⁹¹ Elutriation methods possess the distinct advantage that the effects of incomplete disintegration in the preliminary preparation of the sample are reduced to a minimum by the continual agitation of the particles in the current. However, these methods also have serious disadvantages. The postulated uniform rising current is essentially unattainable. Friction along the walls of the vessel may result in a velocity at the center twice as great as the average velocity.⁹² Turbulent flow, induced by the conical shape of the vessel (so shaped to prevent fine grains from escaping below the inlet of the current), may completely vitiate the results.⁹³ Elutriation, without some centrifugal apparatus to speed up sedimentation, is essentially inapplicable to fine clays, for the low current velocities required are attainable only with great difficulty. A minor objection is that a large sample is necessary if the sample is to be separated into many sizes. The methods require special apparatus, carefully regulated and manipulated, in order to maintain currents of known velocity rising through the suspension. Especially is this true if a battery of tubes is used to separate grains of several different sizes at one time. However, medium-grained silts can be separated into a few size fractions very satisfactorily by elutriation.

⁸⁹ Hilgard, E. W., On the silt analysis of soils and clays: *Am. Jour. Sci.*, 3d ser., vol. 6, pp. 288-296, 333-339, 1873. Baker, H. A., On the investigation of the mechanical composition of loose arenaceous sediments by the method of elutriation, with special reference to the Thanet beds on the southern side of London Basin: *Geol. Mag.*, vol. 57, pp. 327-329, 1920. Holmes, A., Petrographic methods and calculations, pt. 1, pp. 209-215, 1921.

⁹⁰ Lowry, T. M., A new elutriator for rapid use: *Faraday Soc. Trans.*, vol. 18, pp. 32-33, 1922.

⁹¹ Mieczynski, T., Über eine Methode der mechanischen Bodenanalyse und der Bodendispersion: IV^{tes} Conférence Internat. pédologie [Rome] *Actes*, vol. 2, pp. 102-116, 1926.

⁹² Baker, H. A., *op. cit.*, p. 328.

⁹³ Hilgard, E. W., *op. cit.*, p. 291.

DECANTATION METHODS

Simple decantation methods⁹⁴ are particularly useful because they require no special apparatus and because the degree of disintegration and separation attained can be easily determined by simple examination at any stage in the analysis. However, decantation is very laborious. Many decantations are necessary in order to attain a satisfactory separation even into two sizes,⁹⁵ and each additional size requires an additional series of decantations. This method was used in determining the percentage of sand-sized material in the bentonite samples from the Black Hills region,⁹⁶ but it seemed too laborious to use for determining the mechanical composition of this group of shale, mudstone, and marl samples.

"SEDIMENTATION" METHODS

In the "sedimentation" methods the rate at which particles settle out of the suspension is determined by noting (1) the increasing weight or volume of the settled sediment, (2) the decreasing density of the suspension or weight of suspended material at some depth, or (3) the decreasing turbidity of the suspension.

Weight or volume of settled sediment.—Perhaps the simplest of these methods is the one in which the volume of sediment that accumulates from a thoroughly mixed suspension at the bottom of a long graduated tube is read at frequent intervals.⁹⁷ The observed rate of accumulation is plotted, and the proportions of grains of different settling velocities are determined graphically. This simple device is free from some of the errors inherent in other "sedimentation" methods, but, in addition to being liable to some more or less preventable errors, such as the effect of the walls in a narrow tube upon settling velocities, the method is open to two serious objections. First, the bulk volume of accumulated sediment depends largely upon the closeness of packing of the grains, and this closeness of packing varies greatly with the size of the grains, the time the sediment stands, and the load of overlying sediment. (See pp. 16, 34-35.) All these variables, which affect the volume of accumulated sediment, change under the conditions of Werner's method, and the calibration of his curve is therefore exceedingly difficult. Second, the method has practical as well as theoretical difficulties. In order to obtain a

⁹⁴ Fletcher, C. C., and Bryan H., *op. cit.*, pp. 10-11. Goldman, M. I., Petrography and genesis of the sediments of the Upper Cretaceous of Maryland: Maryland Geol. Survey, Upper Cretaceous, pp. 113-120, 1916. Sauramo, Matti, *op. cit.*, pp. 16-17.

⁹⁵ Wentworth, C. K., Methods of mechanical analysis of sediments: Iowa Univ. Studies, vol. 11, No. 11, pp. 41-42, 1926.

⁹⁶ Rubey, W. W., Cretaceous and Cenozoic formations on the northwest flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [In preparation].

⁹⁷ Werner, Donovan, A simple method of obtaining the size distribution of particles in soils and precipitates: *Faraday Soc. Trans.*, vol. 21, pp. 381-394, 1926.

readable volume of accumulating sediment and to avoid a too concentrated suspension (p. 18), the settling tube should be 3 to 10 feet long. With so great a settling distance, even the coarsest clay particles require days to settle to the bottom of the tube, and a long period of settling increases other errors (pp. 18, 28).

Results that are probably more reliable can be obtained by measuring the weight instead of the bulk volume of the accumulated sediment. In the Odén method of continuous weighing⁹⁸ the sediment accumulates on a submerged weighing pan suspended from a balance. Elaborate devices to measure this increasing weight automatically have been superimposed upon the basically simple method.⁹⁹ Like other "sedimentation" methods, this one is least accurate for the coarser grains, which fall rapidly and set up vertical currents, and for very fine grains, whose settling velocities are disturbed by convection currents¹ due to changes in temperature. The desirability of using rather brief settling periods (see pp. 18, 28) justifies the convenience of a short settling distance.

The fundamental assumption of this method—that the particles fall vertically and therefore that those which accumulate upon the weighing pan are typical of the rest—has been questioned. It has been noted that usually only 65 to 95 per cent of the expected sediment (as calculated from the proportion of the total volume of suspension that was originally directly above the pan) actually falls upon the pan and that the higher the pan above the bottom of the settling vessel the greater this discrepancy.² Shaw and Winterer attribute this effect to a mutual repulsion among the deflocculated particles which causes them to move toward the walls; these writers conclude that the method is probably inaccurate for particles smaller than silt. Coutts and Crowther state that the absence of sedimentation under the pan sets up currents that deflect particles from a vertical fall.

However, as discussed more fully on page 29, this loss might possibly be explained in other ways,

⁹⁸ Odén, Sven, Eine neue Methode zur mechanischen Bodenanalyse: Internat. Mitt. Bodenkunde, Band 5, pp. 257–311, 1915; On the size of particles in deep-sea deposits: Roy. Soc. Edinburgh Proc., vol. 36, pp. 219–236, 1916. Vaughan, T. W., Abstract of Sven Odén's work on the determination of the effective radius of particles by their rate of settling in water: Nat. Research Council Comm. on Sedimentation Rept. [Apr. 18, 1923], pp. 41–49, 1923.

⁹⁹ Svedberg, T., and Rinde, H., The determination of size particles in disperse systems: Am. Chem. Soc. Jour., vol. 45, pp. 943–954, 1923. Coutts, J. R. H., Crowther, E. M., Keen, B. A., and Odén, Sven, An automatic and continuous recording balance: Roy. Soc. London Proc., vol. 106 A, pp. 33–51, 1924. Odén, Sven, The size distribution of particles in soils and the experimental methods of obtaining them; a review: Soil Sci., vol. 19, pp. 1–36, 1925.

¹ Fisher, R. A., and Odén, Sven, The theory of the mechanical analysis of sediments by means of the automatic balance: Roy. Soc. Edinburgh Proc., vol. 44, pp. 98–115, 1924.

² Coutts, J. R. H., and Crowther, E. M., A source of error in the mechanical analysis of sediments by continuous weighing: Faraday Soc. Trans., vol. 21, pp. 374–380, 1926. Shaw, C. F., and Winterer, E. V., A fundamental error in mechanical analysis of soils by the sedimentation method: First Internat. Cong. Soil Sci. (Washington), Commissions 1 and 11, Abstracts of Proc., pp. 5–9, 1927.

and some of the other possible explanations would justify the inclusion of the loss with the finest fraction. Whatever the correct explanation of the loss, Coutts and Crowther's data indicate that approximately equal quantities of coarse and fine particles are lost. Therefore the fundamental assumption that those particles which accumulate upon the pan are typical of the rest still seems justified, for if large, intermediate, and small particles are equally affected, the actual amount falling upon the pan is of less importance.

The Odén method has been criticized on the ground that its accuracy depends upon the degree of dispersion of the clay.³ This criticism applies equally to other "sedimentation," elutriation, decantation, sieving, and even to microscopic methods. The dispersion or disintegration of the original sample is a problem of preparation rather than of methods of measurement. Although the errors arising from incomplete dispersion do not form a specific criticism of the Odén method of continuous weighing, they must of course be kept in mind in deciding upon the degree of refinement justifiable in any method of measurement.

A somewhat similar method is one in which the sediment that accumulates near the bottom of the settling vessel is continuously removed for separate weighing.⁴

Density of suspension.—The decreasing density of the suspension or weight of suspended sediment may be measured (*a*) by direct weighing of the suspended matter at different depths as sampled with a pipette, (*b*) by the buoyant effect of the suspension upon a hydrometer, and (*c*) by a manometer or by the height of a fluid column of known density that balances the weight of a certain part of the suspension. A serious objection to all these methods is that they require maximum allowable concentrations of the suspension (see p. 18) in order to afford measurable quantities of suspended matter.

Pipette method: For direct weighing the suspension may be sampled at different depths and at different times with a pipette.⁵ The method requires no special apparatus, and if sufficiently large volumes of suspension (and therefore long settling times) are used,

³ Ormandy, W. R., discussion on "Colloidal phenomena": Faraday Soc. Trans., vol. 17, pp. 366–367, 1922. Trowbridge, A. C., discussion on "Sediments and sedimentation": Nat. Research Council, Comm. on Sedimentation, Rept. [Apr. 18, 1923], pp. 54–55, 1923. Gile, P. L., Middleton, H. E., Robinson, W. O., Fry, W. H., and Anderson, M. S., Estimation of colloidal material in soils by adsorption: U. S. Dept. Agr. Bull. 1193, pp. 7, 35, 1924.

⁴ Schloesing, T., sr., Sur l'analyse mécanique des sols: Compt. Rend., vol. 136, pp. 1608–1613; vol. 137, pp. 369–374, 1903.

⁵ Jennings, D. S., Thomas, M. D., and Gardner, W., A new method of mechanical analyses of soils: Soil Sci., vol. 14, pp. 485–499, 1922. Robinson, G. W., A new method for the mechanical analysis of soils and other dispersions: Jour. Agr. Sci., vol. 12, pp. 306–321, 1922. Krauss, G., Ergänzender Bericht über eine * * * neue Methode der mechanischen Bodenanalyse: Internat. Mitt. Bodenkunde, Band 13, pp. 147–160, 1923.

if samples are drawn off at frequent intervals, and if proper allowance is made for the height of removed water, a very accurate distribution curve of the weights of particles with different settling velocities can be computed. However, the method is at best slow and laborious compared with some of the other "sedimentation" methods. A modification of this method is a settling tube provided with stopcocks from which the suspension can be drawn off at different depths.⁶

Hydrometer method: Measurements of the decreasing weight of suspended matter have been made with a plummet of known volume suspended from a balance at a constant depth in the suspension⁷ and with a sensitive floating hydrometer.⁸ Sweeping claims have been made for these methods, but accurately measurable differences in the density of a settling suspension are attained only if the suspension is very highly concentrated, and under these conditions the settling velocities are at first abnormally slow. (See p. 18.) The floating hydrometer sinks deeper as the particles settle, and hence, as it measures the weight of particles in a column of constantly changing height, it is exceedingly difficult to calibrate. Particles accumulate on the plummet of either a floating or a suspended hydrometer and weight it down, thus giving readings that indicate erroneously rapid settling velocities. In some preliminary tests the writer obtained readings at the beginning of the settling period that were much higher than the true average specific gravity of the suspension. This discrepancy, which was also noted by Schurecht,⁹ may be the result of rising currents generated by the rapidly falling larger particles. In this as in other "sedimentation" methods that start with a thoroughly mixed suspension, the data, even when corrected for other errors, must still be recalculated to allow for the amount of material that falls from intermediate depths. (See pp. 19, 24-25.) The errors in these hydrometer methods seem to be so large and numerous that interpretation of the results into a size or settling-velocity curve is nearly if not quite impossible.

Manometer method: The decreasing hydrostatic pressure in a suspension may be measured by the height

to which a fluid of known density rises in a manometer or side tube attached to the settling vessel.¹⁰ With moderate or dilute concentrations the fluid in the side tube rises only a short distance above the suspension, and so can not be read very accurately. Various modifications have been proposed to increase the accuracy of the method. For example, the vertical displacement can be read more closely if the side tube is bent over nearly horizontal,¹¹ and a liquid of lower specific gravity in the side tube can be read more accurately.¹² Readings may be continued for days or weeks, if necessary, without fear of damaging the instrument, and, in this respect at least, these manometer methods are superior to the methods that involve continuous weighing. In fact, preliminary tests lead the writer to believe that the general principle of manometer measurement may eventually be developed into the most thoroughly satisfactory method for mechanically analyzing fine-grained sedimentary rocks. However, present methods based on this principle are especially subject to errors caused by small variations in temperature¹³ and by evaporation of the liquid in the side tube; also the initial shaking of the suspension to attain the uniform distribution of particles is difficult because of the necessary side tubes.

Turbidity of suspension.—For theoretical elegance and practical complexity no other method can compare with those in which the decreasing turbidity of the suspension is measured. The opacity of a suspension at different heights and at different times can be measured quantitatively with photometric apparatus, and from the amount of light absorbed or reflected the concentration of the suspension and the size distribution of particles can be calculated. The method has been used and verified by microscopic examinations.¹⁴ However, calculation of particle size from the turbidity of a suspen-

¹⁰ Wiegner, G., über eine neue Methode der Schlammanalyse: Landw. Vers. Sta., Band 91, pp. 41-79, 1918.

¹¹ Kraemer, E. O., and Stamm, A. J., A new method for the determination of the distribution of size of particles in emulsions: Am. Chem. Soc. Jour., vol. 46, pp. 2709-2718, 1924. Kelly, W. J., Determination of distribution of particle size: Ind. and Eng. Chemistry, vol. 16, pp. 928-930, 1924.

¹² Odén, Sven, The size distribution of particles in soils and the experimental methods of obtaining them: Soil Sci., vol. 19, pp. 25-30, 1925. See also Zunker, I. F., Die Bedeutung und Bestimmung der spezifischen Oberfläche des Bodens: IV^{ème} Conférence internat. pédologie [Rome] Actes, vol. 2, pp. 238-249, 1926; Crowther, E. M., A manometric apparatus for the direct determination of summation percentage curves in mechanical analysis: First Internat. Cong. Soil Sci. (Washington), Commissions I and II, Abstracts of Proc., pp. 13-18, 1927.

¹³ Odén, Sven, Soil Sci., vol. 19, pp. 23, 25, 1925.

¹⁴ Svedberg, T., and Nichols, Z. B., Determination of size and distribution of size of particles by centrifugal methods: Am. Chem. Soc. Jour., vol. 45, pp. 2910-2917, 1923. Svedberg, T., and Rinde, H., The ultra centrifuge, a new instrument for the determination of size and distribution of size of particle in amicroscopic colloids: Idem, vol. 46, pp. 2677-2693, 1924. Stamm, A. J., and Svedberg, T., The use of scattered light in the determination of the distribution of size of particles in emulsions: Idem, vol. 47, pp. 1582-1596, 1925.

⁶ Trnka, R., Notes sur l'analyse mécanique du sol: IV^{ème} Conférence internat. pédologie [Rome] Actes, vol. 2, pp. 116-122, 1926.

⁷ Pratolongo, U., Sull' analisi fisico-meccanica dei terreni: Staz. Sper. Agr. Ital., vol. 50, pp. 117-166, 1917; cited by Odén, Sven, Soil Sci., vol. 19, pp. 21-22, 1925. Schurecht, H. G., Sedimentation as a means of classifying extremely fine clay particles: Am. Ceramic Soc. Jour., vol. 4, pp. 812-821, 1921. Mleczynski, T., Über eine Methode der mechanischen Bodenanalyse und der Bodendispersion: IV^{ème} Conférence internat. pédologie [Rome] Actes, vol. 2, pp. 102-116, 1926.

⁸ Bouyoucos, G. J., Estimation of the colloidal material in soils: Science, new ser., vol. 64, p. 362, 1926; A rapid method for mechanical analysis of soils: Idem, vol. 65, pp. 549-551, 1927; Directions for determining the colloidal material of soils by the hydrometer method: Idem, vol. 66, pp. 16-17, 1927.

⁹ Schurecht, H. G., op. cit., p. 820.

sion is exceedingly difficult, because opacity depends not only upon the number of particles in suspension and the intensity and wave length of the light used but also upon the size of the particles, the difference between the refractive indices of the particles and the liquid, the color and transparency of the particles,¹⁵ and the features of their surface that affect the amount of light reflected. Hence, a separate calibration is necessary for each substance examined. Morison,¹⁶ who attempted to apply the method to soils, admits that interpretation of particle diameters from determinations of turbidity is virtually impossible with our present knowledge. In view of the elaborateness of the apparatus required and the uncertainty of the results obtained, it seems fairly clear that the method is not a suitable one for the mechanical analysis of fine-grained sedimentary rocks.

PRESENTATION OF DATA

Just as there are different ways of preparing the samples and of determining the proportions of large and small grains, so there are different ways of stating the results. Mechanical analyses are not an end in themselves but are made to be used for some particular purpose, and therefore the form of presentation may be quite as important as the preparation and measurement. Some methods of presentation of the data are simple but crude; others give a more complete statement but are difficult to interpret. A sample may contain 75 per cent by weight of material finer than sand. The same sample may consist of 1 per cent of gravel, 24 per cent of sand, 60 per cent of silt, and 15 per cent of clay; and each of these groups may be further subdivided into coarse, medium, and fine fractions. As the number of subdivisions is thus increased, the statement of the composition becomes more complete but more difficult to visualize. An elaborate mathematical equation might be written to express the complete size distribution, but it would be laborious to formulate and probably more detailed than the accuracy of the data would warrant, and its significance would be difficult to grasp.

DIAGRAMS

Graphic methods of presenting the data undoubtedly give the largest amount of information in a readily understandable form. Hilgard¹⁷ illustrated his mechanical analyses of soils by photographs of the different size separates lined up side by side in test tubes. The same effect can be obtained more

easily by plotting the weights of each portion in vertical columns in the order of grain size.¹⁸ These pyramidal diagrams, or histograms, as they have been called,¹⁹ have been widely used by geologists. They are valuable in that they give a simple visual picture of the proportions of particles having diameters between several size limits—that is, the size and degree of sorting of a sample. However, they are unsatisfactory in that the size of the limits which happen to be used affects the form of the resulting graph greatly.²⁰ Increasing the number of limits and so narrowing the range of sizes between limits decreases this artificial effect and eventually gives a distribution or frequency curve²¹ of the sizes that is complete, but this curve, like the mathematical equation, is tedious to derive either by experiment or by calculation.

A second type of diagram that has been much used by geologists and engineers is called the cumulative curve and shows the summed percentages of grains of different sizes.²² Particle diameters may be so plotted as to increase toward either the right or the left of the diagram. If they increase toward the right, the diagram represents the percentages by weight of grains smaller than the given diameters; if toward the left, it shows the percentages larger than the given diameters. Plotting the larger diameters at the left and the smaller at the right seems to have been the more used and is much the easier of the two procedures if "sedimentation" methods of analysis are employed. These cumulative curves are valuable because, in representing natural granular mixtures such as sedimentary rocks and soils, they are commonly smooth curves and can be interpolated with fair accuracy from a small number of experimentally determined points.²³ That is to say, the size limits that may have happened to be used in measurement scarcely affect the result; hence the cumulative curves express the size distribution of a sample rather completely and with a minimum of effort. However, until these curves become familiar they are not as easily interpreted as the pyramidal diagrams.

In diagrams of both types the particle diameters may be plotted directly, or the logarithms of the

¹⁵ Stutz, G. F. A., and Pfund, A. H., A relative method for determining particle size of pigments: *Ind. and Eng. Chemistry*, vol. 19, pp. 51-53, 1927.

¹⁶ Morison, C. G. T., The effect of light on the settling of suspensions: *Roy. Soc. London Proc.*, vol. 108 A, pp. 280-284, 1925.

¹⁷ Hilgard, E. W., *Soils*, pp. 95-96, Macmillan Co., 1906.

¹⁸ Goldman, M. I., Petrography and genesis of the sediments of the Upper Cretaceous of Maryland: *Maryland Geol. Survey, Upper Cretaceous*, pp. 122-123, 169-170, 1916.

¹⁹ Wentworth, C. K., Methods of mechanical analysis of sediments: *Iowa Univ. Studies*, vol. 11, pp. 46-51, 1926.

²⁰ Dake, C. L., The problem of the St. Peter sandstone: *Missouri Univ. School of Mines and Metallurgy Bull.*, vol. 6, pp. 173-174, 1921. Wentworth, C. K., *op. cit.*, pp. 47, 52.

²¹ Odén, Sven, On the size of particles in deep-sea deposits: *Roy. Soc. Edinburgh Proc.*, vol. 36, pp. 219-236, 1916.

²² Slichter, C. S., The motions of underground waters: *U. S. Geol. Survey Water-Supply Paper* 67, p. 23, 1902. Dake, C. L., *op. cit.*, pp. 156-157. Holmes, A., Petrographic methods, pt. 1, pp. 215-226, 1921. Wentworth, C. K., *op. cit.*, pp. 49-51.

²³ Robinson, G. W., The form of mechanical composition curves of soils, clays, and other granular substances: *Jour. Agr. Sci.*, vol. 14, pp. 626-633, 1924.

diameters may be used.²⁴ If the diameters are plotted directly, poorly sorted rocks require very large diagrams, but the logarithms of the diameters can be plotted much more compactly. Furthermore, whether the settling velocity or the velocity of a current competent to move a grain is considered, the logarithms are probably of greater geologic significance than the actual diameters. The difference between diameters of 0.2 and 0.1 millimeter and between diameters of 10.2 and 10.1 millimeters is in each case 0.1 millimeter, but the proportional difference between volumes, weights, or settling velocities of the two smaller grains is tremendously greater. For these two reasons all systems of size classification of rock particles known to the writer are at least crudely logarithmic or based on ratio of sizes. For example, in Udden's classification,²⁵ boulders have diameters from 256 to 16 millimeters, gravel from 16 to 1 millimeter, sand from 1 to $\frac{1}{16}$ millimeter, and silt from $\frac{1}{16}$ to $\frac{1}{256}$ millimeter—that is, the ratio of size limits is 16 to 1. For the same reason most geologists have used some sort of logarithmic scale in plotting the particle diameters on graphs showing mechanical analyses.

A third type of diagram is that in which the sample is divided into three fractions, such as sand, silt, and clay, and plotted on a triangular diagram.²⁶ This method omits much valuable information and is normally used only for special purposes.

In the methods thus far considered the proportion by weight of grains of different sizes is used. It would be more consistent with a classification based upon particle diameters to express the percentages by volume, especially if it is true that the smaller particles in soils and rocks commonly have a lower specific gravity than the larger particles and if the significant physical properties are the result of size rather than of weight.²⁷ However, most of the methods of mechanical analysis that have been proposed measure the weight and not the volume of the different sizes (but see pp. 19, 20) and without separate determinations of the specific gravity of the different sizes conversion of the data into volumes is impossible.

SIZE LIMITS OF SAND, SILT, AND CLAY

It is desirable to have names for the different size fractions separated, as convenient names help greatly to classify the material in a reader's mind. A rock composed chiefly of gravel is very different from one composed chiefly of clay; even "fine sand" carries a very different picture from "coarse sand." Unfor-

tunately, however, conceptions differ as to the precise limits that separate, let us say, clay from silt.

Of the many classifications that have been proposed,²⁸ there are two that are widely used. Atterberg's system²⁹ (boulders, more than 2,000 millimeters; blocks, 2,000 to 200 millimeters; pebbles, 200 to 20 millimeters; gravel, 20 to 2 millimeters; sand, 2 to 0.2 millimeter; powder sand, 0.2 to 0.02 millimeter; silt, 0.02 to 0.002 millimeter; clay, less than 0.002 millimeter) is based upon those diameters at which notable changes in physical behavior, such as mobility in storm waves, capillary movement of ground water, and coagulation by salt water, become noticeable. This classification system, with each group divided into three fractions by limiting diameters of 20, 10, 5, 2 millimeters, etc., and with the addition of a colloid group (less than 0.0002 millimeter), is widely used by soil scientists³⁰ and European geologists. The other classification is that proposed by Wentworth,³¹ who ascertained the actual usage of size terms by several score geologists and as a result recommended a system very similar to that used by Udden,³² cited above. The Atterberg and Wentworth-Udden systems are similar but by no means identical.

EXACT RELATION BETWEEN SETTLING VELOCITY AND DIAMETER OF IRREGULAR PARTICLES UNKNOWN

The Stokes law is not precise when applied to sand-sized or nonspherical particles (pp. 17-18) and therefore the settling velocities of rock particles determined by elutriation, decantation, and "sedimentation" methods can not be converted into precise particle diameters. The settling velocities of different-sized particles have been determined experimentally by many investigators, but these determinations show much variation. The Atterberg scale³³ of settling velocities (the time required for a particle to settle 10 centimeters) is widely used by soil scientists, but it differs greatly from the settling velocities found by other investigators³⁴ and from average diameter-velocity relations recommended by Holmes³⁵ and Wentworth.³⁶

²⁸ Briggs, L. J., Martin, F. O., and Pearce, J. R., The centrifugal method of mechanical analysis: U. S. Dept. Agr. Bur. Soils Bull. 24, pp. 32-33, 1904. Wentworth, C. K., A scale of grade and class terms for clastic sediments: Jour. Geology, vol. 30, p. 384, 1922. Odén, Sven, The size distribution of particles in soils and the experimental methods of obtaining them: Soil Sci., vol. 19, p. 2, 1925.

²⁹ Atterberg, A., Die rationelle Klassifikation der Sande und Kiese: Chem. Zeitung, Jahrgang 29, pp. 195-198, 1905.

³⁰ Krauss, G., Beitrag zur mechanischen Bodenanalyse: IV^{ème} Conférence Internat. pédologie [Rome] Actes, vol. 2, pp. 122-126, 1926.

³¹ Wentworth, C. K., A scale of grade and class terms for clastic sediments: Jour. Geology, vol. 30, pp. 377-392, 1922.

³² Udden, J. A., op. cit.

³³ Atterberg, A., op. cit., p. 198.

³⁴ For a comparison of several sets of determinations see Mohr, E. C. J., Mechanische Bodenanalyse: Dept. agr. Indes Néerlandaises Bull. 41, p. 16, Bultenzorg, 1910.

³⁵ Holmes, A., op. cit., p. 207.

³⁶ Wentworth, C. K., Methods of mechanical analysis of sediments: Iowa Univ. Studies, vol. 11, p. 41, 1926.

²⁴ Bøggild, O. B., Samples of the sea floor: Copenhagen Univ. Min. and Geol. Mus. Contr. Mineralogy, No. 3, p. 34, 1903.

²⁵ Udden, J. A., Mechanical composition of clastic sediments: Geol. Soc. America Bull., vol. 25, pp. 655-744, 1914.

²⁶ Holmes, A., Petrographic methods, pt. 1, pp. 226-230, 1921.

²⁷ Biéler-Chatelan, T., Constitution volumétrique des sols en place: IV^{ème} Conférence Internat. pédologie [Rome] Actes, vol. 2, pp. 187-208, 1926.

To avoid this uncertainty, Odén converts the observed settling velocity into an "equivalent radius"³⁷—the radius of a perfect sphere that theoretically would settle at the same rate as the actual particle. Robinson³⁸ considers the uncertainties due to shape and the possible gel envelopes surrounding the smaller particles so great that he recommends using the settling velocities themselves (at some standard temperature) or their logarithms instead of the supposed diameters.

GRAPHIC SOLUTION OF "ACCUMULATION" AND CONCENTRATION CURVES

Many of the "sedimentation" methods of mechanical analysis start with a thoroughly mixed suspension and measure either the progressive increase in weight or volume of sediment that accumulates at the bottom of a tube or the progressive decrease in concentration of the suspension. The accumulated sediment at any given time consists of two parts—(1) large particles whose settling velocities are so great that even those particles which started falling from the very top of the suspension have had time to settle to the bottom, and (2) that portion of the smaller, more slowly falling particles which started falling from such intermediate depths in the suspension that they also have had time to settle to the bottom. The amount of the smaller particles mixed with the large ones in the accumulated sediment at any moment must be determined before the true composition of the sample is known. This correction can be calculated either mathematically³⁹ or graphically.⁴⁰ The necessity for this mathematical correction is a feature of most "sedimentation" methods of mechanical analyses which has greatly impressed some geologists and which, on the other hand, has been overlooked by some workers in other sciences.

The graphic method of making the correction is much simpler and quicker than the mathematical calculation, and for most data it is probably quite as accurate. It depends upon the fact that in a thoroughly mixed suspension particles of any one size, being uniformly distributed, start falling from

all heights and accumulate on the bottom at a uniform rate until the last particles of this size (those that started at the very top of the suspension) have settled to the bottom; that is, if the suspension is sufficiently dilute at the outset so that changes in the specific gravity and viscosity of the liquid are negligible. If particles of two or more sizes are present in the suspension, the rate of their total or joint accumulation is a steady one until all of the largest size (A) have fallen to the bottom, and then the total rate of accumulation decreases abruptly. The rate of accumulation immediately after all the size A particles have settled is the rate at which the particles smaller than size A had been accumulating since the beginning—that is, the previous joint rate was faster because the particles of size A were also settling. When the rate of accumulation of the smaller particles is thus ascertained the total amount of the smaller particles that had settled at any time can be determined by multiplication—that is, the total amount of smaller particles that accumulated with those of size A is the product of the rate at which they had been accumulating since the beginning and the time required for all the particles of size A to settle. Similarly, the amount of still smaller particles that accumulate with those of size B is the product of their rate of accumulation and the time required for those of size B to settle, and so on.

The different rates of accumulation at the times when particles of different sizes finish falling, which are required for this computation, can be determined by rather laborious mathematics. However, plotting the time and the weight of sediment accumulated at the bottom of a suspension yields a curve that shows graphically a constantly decreasing rate of accumulation. (See pl. 1, A.) Each bend in the curve represents the time at which the last particles of some particular size reached the bottom. The slope of the curve an infinitely short time after it passes this bend gives the rate of accumulation of the smaller particles that are still settling and therefore the rate at which these smaller particles have been accumulating since the beginning.

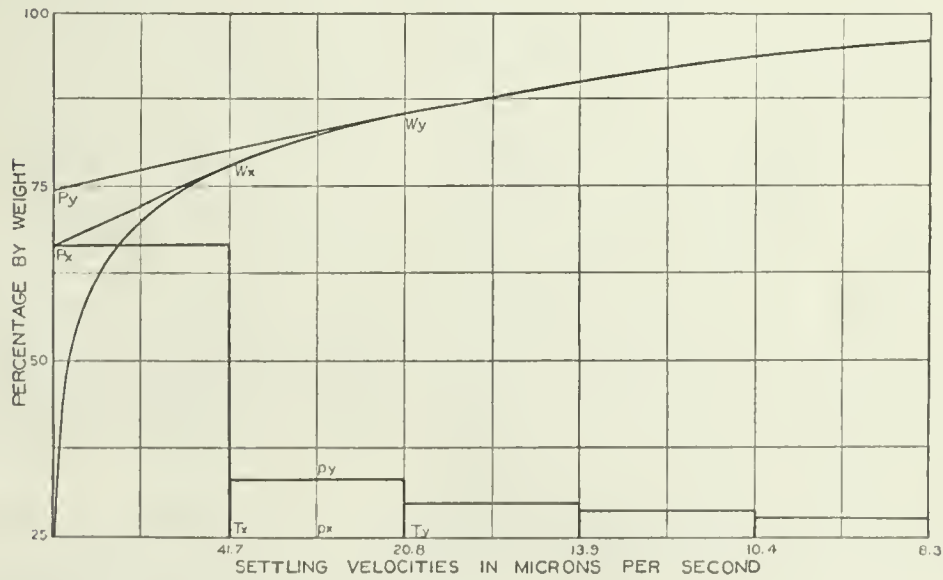
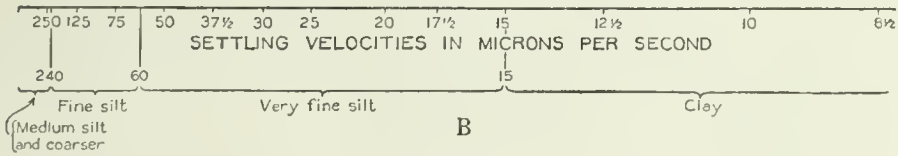
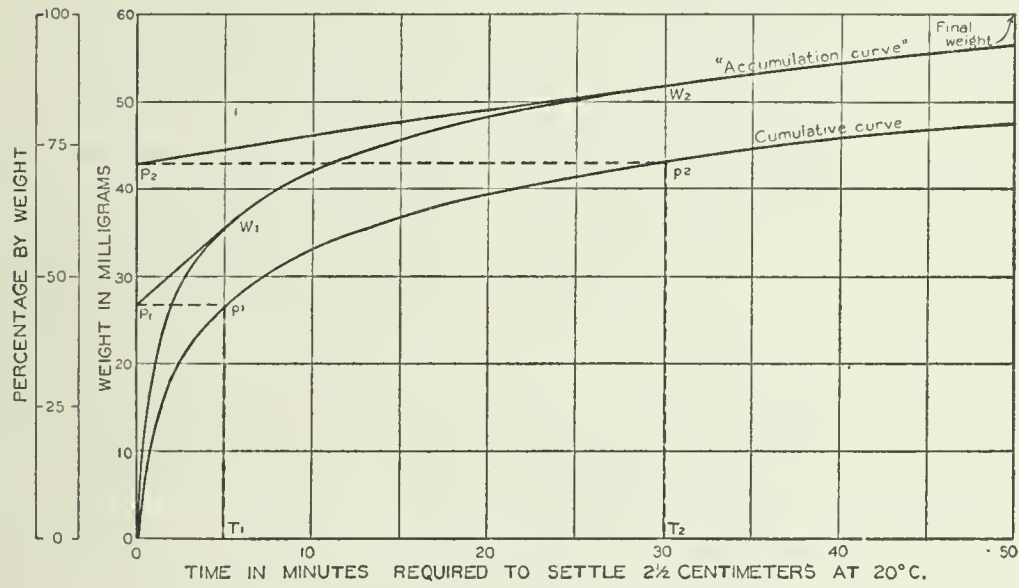
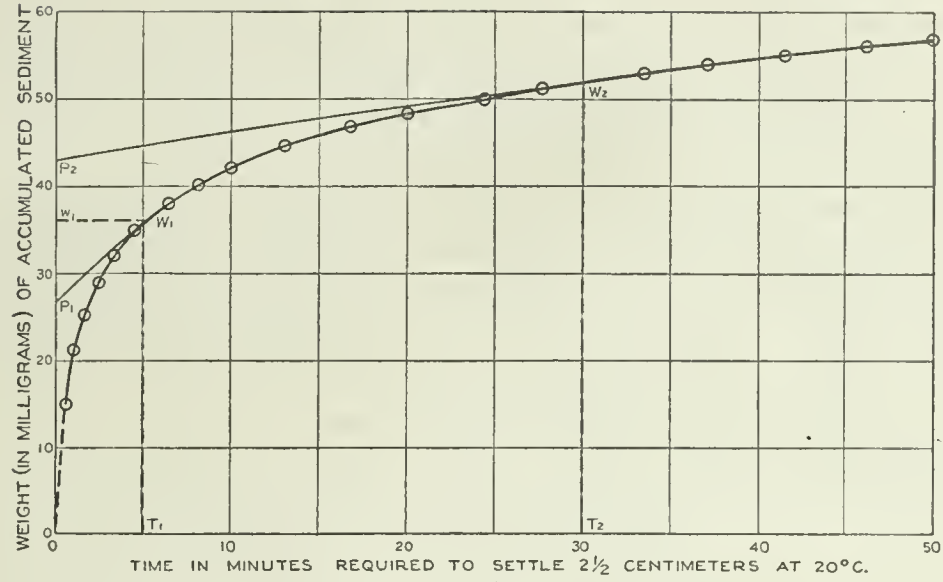
Hence, by drawing the straight line $W_1 P_1$ (pl. 1, A) tangent to one of the bends on the curve until it intercepts the vertical axis, and by putting on this axis w_1 equal to W_1 , the weight of smaller particles that settled with the particular size indicated by the bend is determined. That is, this weight, represented by the line $w_1 P_1$, equals the rate of accumulation of these particles (or, stated in mathematical terms, the derivative or slope of the curve at this point, or the tangent of the angle $w_1 W_1 P_1$) multiplied by the time T_1 . Then P_1 , the weight of larger particles that required time T_1 , or less, to settle from the very top

³⁷ Odén, Sven, The size distribution of particles in soils and the experimental methods of obtaining them: *Soil Sci.*, vol. 19, p. 3, 1925.

³⁸ Robinson, G. W., On certain regularities in the mechanical composition of soils and other granular substances: *IVème Conférence internat. pédologie [Rome] Actes.* vol. 2, pp. 180–187, 1926; The grouping of fractions in mechanical analysis: *First Internat. Cong. Soil Sci. (Washington), Commissions I and II, Abstracts of Proc.*, pp. 1–2, 1927.

³⁹ Odén, Sven, On the size of particles in deep-sea deposits: *Roy. Soc. Edinburgh Proc.*, vol. 36, pp. 227–230, 1916.

⁴⁰ Svedberg, T., and Rinde, H., The determination of the distribution of size particles in disperse systems: *Am. Chem. Soc. Jour.*, vol. 45, p. 947, 1923. Schramm, E., and Scripture, E. W., jr., The particle analysis of clays by sedimentation: *Am. Ceramic Soc. Jour.*, vol. 8, pp. 243–252, 1925. Werner, Donovan, A simple method of obtaining the size distribution of particles in soils and precipitates: *Faraday Soc. Trans.*, vol. 21, p. 342, 1926. Calbeck, J. H., and Harner, H. R., Particle size and distribution by sedimentation method: *Ind. and Eng. Chemistry*, vol. 19, pp. 59–60, 1927.



METHOD OF DERIVING TRUE MECHANICAL COMPOSITION OF A GRANULAR MIXTURE FROM SUCCESSIVE WEIGHTS OF ACCUMULATED SEDIMENT, WITH GRAPHIC DETERMINATION OF CUMULATIVE CURVE AND PYRAMIDAL DIAGRAM FROM "ACCUMULATION CURVE"

of the suspension, equals the total weight at the time T_1 minus the weight of the smaller particles. Stated mathematically, $P = W - T \frac{dW}{dT}$. Where P = corrected weight, W = uncorrected weight, T = time, and $\frac{dW}{dT}$ = change of W with respect to change of T . Similarly drawing a straight-line tangent from W_2 determines P_2 , the weight of larger particles that had completely settled out of the suspension in the time T_2 , and so on along the curve.

There yet remains the task of presenting the data in some readily usable form. P_2 consists of P_1 plus those smaller particles that completely settled out of the suspension in the time T_2 . Now from the known total depth of the suspension the different times, T_1 , T_2 , etc., required for complete settling of particles of different sizes can be converted into settling velocities for the respective sizes. Also, from the final total weight of accumulated sediment the percentage of sediment with settling velocities greater than any chosen velocity can be calculated. Therefore, by changing the vertical and horizontal axes of the graph from weight and time to percentage and velocity and by plotting p_1 equal to P_1 at time T_1 , p_2 equal to P_2 at time T_2 , etc. (pl. 1, B), the "accumulation curve"⁴¹ can very readily be converted into a cumulative curve showing the total percentage of particles that fall more rapidly than certain velocities. If the diameters corresponding to the different settling velocities are known, the horizontal axis of the graph can be changed from velocities to diameters, and the cumulative curve of settling velocities then becomes a cumulative curve of particle sizes or a diagram showing the percentage of particles larger than any chosen diameters.

A pyramidal diagram or histogram of the sample can be drawn with very little additional work. In section C of Plate 1, P_y minus P_x is the weight of sediment with settling times between T_x and T_y . Therefore the column $p_x p_y$, with a height equal to the line $P_x P_y$, represents the percentage of the sample between the two settling velocities or sizes indicated.

The foregoing discussion applies to the increasing weight of accumulated sediment at the bottom of a suspension. For the decreasing concentration of the suspension, determined by some method of measuring density or turbidity, the method of calculation would be the same, and the concentration curve to be used would be the "accumulation curve" inverted.

PROCEDURE ADOPTED AND RESULTS

Preparation.—After some preliminary tests upon several methods of disintegration, such as boiling in water, crushing with a wooden mallet, kneading with the fingers, digestion in dilute hydrochloric acid, and

continuous agitation in water for several hours, it was decided to adopt a long period of soaking in slightly ammoniacal water, with frequent and vigorous shaking and occasional rubbing with a rubber pestle.

Clean-surfaced, nearly equidimensional fragments of the shale, weighing 1 gram at a relative humidity of 65 per cent on a balance accurate to 0.2 milligram, were placed in Florence flasks with 150 cubic centimeters of tap water and 1 cubic centimeter of 25 per cent ammonia solution. These half-filled flasks were tightly stoppered and shaken vigorously for a few seconds almost every day for eight weeks. After one month's soaking and at 1-week intervals thereafter, the fragments of mud were rubbed gently with a rubber-tipped pestle.

Under this treatment some of the samples disintegrated rapidly but others very slowly. The relative rates of disintegration of the different samples showed no relation to the content of carbonate or of water in the air-dried samples, to the porosity, or to the mechanical composition as indicated by microscopic examination and mechanical analyses. In a general way, however, the samples containing the most organic matter and the most pyrite disintegrated most slowly.

Three of the samples that disintegrated most slowly (J, H, and K) colored the water brownish; and as these samples contained relatively large percentages of organic matter, it is probable that the discoloration was caused by organic matter dissolved. Several of the suspensions formed a persistent foam when shaken. Although both the discoloration and the soapiness indicate solution of a part of the rock, neither the viscosity nor the density of the discolored or soapy liquids was changed greatly. After several months' soaking the relative viscosity of the soapiest suspension (measured in a Stormer viscosimeter) was less than 1.05 (showing an increase about equal to that produced by a drop of 1° F. in the temperature of pure water), and the density of the darkest liquid (measured in a Chainomatic specific-gravity balance) was slightly less than 1.001.

The degree of disintegration attained by this treatment was fairly satisfactory. Microscopic examination indicated that after disintegration only 5 to 25 per cent of the grains consisted of aggregates of two or more individual particles. In the final mechanical analyses corrections were made for the observed proportions of these aggregates in the different-size fractions; however, this attempted refinement may not have been worth while, for these aggregates may have settled as such when the mud accumulated on the sea floor.

In an attempt to determine the size distribution of the insoluble particles in the calcareous rocks, three of the more calcareous samples (D, E, and II) were digested in dilute hydrochloric acid (1 part HCl to

⁴¹ Odén, Sven, Roy. Soc. Edinburgh Proc., vol. 36, pp. 227-228, 1916.

25 parts water) for one month. The acid was then decanted, after settling periods that assured the retention of all particles with settling velocities greater than 0.5 micron per second (which is much slower than the settling velocity of large clay particles), and slightly ammoniacal water was added. Microscopic examination showed, however, that this treatment did not disintegrate the insoluble particles very satisfactorily.

Measurement.—After preliminary tests of several methods of measurement, the general principle of continuous weighing was chosen as apparently one of the simplest, quickest, and most practicable ways of obtaining a moderately complete distribution curve of particle sizes. The errors inherent in this method seemed to be no greater than those due to the uncertainty about the degree of flocculation of the clay at the time of deposition, to the possible growth of clay minerals since deposition, and to the incomplete preparatory disintegration of the sample.

A disintegrated sample was washed from its flask into a beaker which was then filled with tap water to 277 cubic centimeters. The contents of the beaker were stirred thoroughly, care being taken to avoid a rotary or centrifugal motion; a platinum pan⁴² was immediately introduced into the suspension, and the sediment that accumulated on this pan was weighed at frequent intervals for about 50 minutes. To check the accuracy of the method, two or more series of weighings were made on many of the samples, and on these the weighing was continued for 1½ to 18 hours.

The pan was suspended by three fine platinum wires 5 centimeters below the upper surface of the liquid and 4 centimeters above the bottom of the beaker; thus the maximum settling distance was known, and the pan was well above the sediment that accumulated on the bottom of the beaker. The diameter of the beaker was 6.3 centimeters and that of the platinum pan 3.6 centimeters, so that the formation of currents between the edges of the pan and the sides of the beaker was probably negligible. The average density of the suspension was only about 1.002 at the beginning of sedimentation and decreased to 1 as sedimentation progressed; therefore errors that might result from using highly concentrated suspensions, such as those caused by interference between particles, flocculation, vertical currents, and increased viscosity and density (see p. 18), were certainly negligible.⁴³ The pan was suspended from a

⁴² In accordance with the usage of some workers an aluminum pan was tried first but was found to be utterly unsatisfactory. Small gas bubbles formed on its surface in the slightly ammoniacal water, thus making accurate weighing impossible.

⁴³ Schloesing, T., sr., *Sur l'analyse mécanique des sols*: Compt. Rend., vol. 136, pp. 1608–1613, 1903. Odén, Sven, *On clays as disperse systems*: Faraday Soc. Trans., vol. 17, p. 334, 1922. Robinson, G. W., *A new method for the mechanical analysis of soils and other dispersions*: Jour. Agr. Sci., vol. 12, pp. 306–321, 1922.

Chainomatic balance to facilitate rapid weighing. This balance was accurate to about 0.8 milligram, and the final weight in water of the sediment that accumulated on the pan ranged from 45 to 100 milligrams, hence the readings were accurate within 2 per cent. At final weighing the sediment on the pan ranged from about 1 to 5 millimeters in thickness, indicating porosities of 95 to 99 per cent. Such thicknesses are a source of considerable error, for when the layer becomes thick fairly large quantities of sediment deposited near the edge of the pan slump off and so are not weighed.

Presentation.—The successive weight readings made during the different series of continuous weighing were plotted on large sheets of coordinate paper and corrected graphically for admixed smaller particles by the method described on pages 24–25. From the known settling distance between the top of the liquid and the pan the different periods of time on the horizontal axis of the graph were readily converted into settling velocities.

However, as a rise in temperature causes a great decrease in the viscosity of water and hence an increase in the settling velocity of particles falling through water, the observed settling velocities of different samples are not comparable unless they are reduced to some one standard temperature. The velocities on the graphs were therefore shifted by the necessary factor to reduce them to a temperature of 20°.

*Decrease in viscosity of water with rising temperature*⁴⁴

	Viscosity of water		Viscosity of water
10° C.....	0.01309	25° C.....	0.00897
15° C.....	.01146	30° C.....	.00803
20° C.....	.01008		

From the final total weight the weights of accumulated sediment at different times were readily converted into percentages. A graph corrected in this manner is a cumulative curve of the percentages by weight of particles that fall more rapidly than certain velocities. (See pl. 1, B and C.) At the left-hand side of this graph high velocities are crowded together, but at the right low velocities are spread wide apart.

Determination of sizes of particles from settling velocities.—The exact diameter of particles can not be calculated from their settling velocities (pp. 23–24). Empirical determinations by different investigators of the diameters of particles with certain settling velocities differ greatly. However, as a check on the method used here, small samples of each of the suspensions were pipetted off at different times, and the largest of the suspended particles in each sample were measured under the microscope. Although the

⁴⁴ Original data from Poiseuille; quoted in Roth, W. A., and Scheel, K., *Physikalisch-chemische Tabellen*, Band 1, p. 136, Berlin, 1923.

diameters of these particles clearly decreased as the time of settling increased, the particles were so irregularly shaped that no exact relation between diameter and velocity could be discovered.

A moment's consideration shows that these discrepancies are not at all surprising. The settling velocity of a particle depends not only upon its effective weight in water but also upon the frictional resistance of the water, which in turn depends upon the shape or surface area of the particle. Among particles of a certain volume and weight but of different shapes, those that are spherical have the smallest surface area and those that are flat or rodlike have much larger areas. The spherical particles therefore fall more rapidly than the flat and fibrous ones.⁴⁵ Furthermore, microscopic measurement of the three dimensions of even those particles with known settling velocities is difficult, for if the particles are free to arrange themselves upon the glass slide, they tend to lie on their flat sides. Hence, for both of these reasons, conflicting results should be expected.

Microscopic examination of samples pipetted out from the suspensions and of thin sections of the shale cut perpendicular to bedding planes shows that in the rocks studied the constituent clay particles are platy and that their thickness is commonly only about one-seventh their length and breadth. On the assumption that the frictional resistance of water is proportional to the total surface area of the falling particles, a disk with these relative dimensions would fall at the same rate as a sphere with the same density and with a diameter only one-third as great, provided that the disk fell with its flat side always directed downward—that it never tipped up on edge and slid through the water. Obviously the platy particles do tip and fall with a zigzag path, and therefore their expected settling velocity should be greater. On the assumption, then, that the particles fall not in some one but in all possible orientations, the frictional resistance of the water would be proportional to the surface area directed vertically downward in the average of all possible orientations—that is, with an inclination of 60° ($\cos 45^\circ \times \cos 45^\circ = \cos 60^\circ$) from the horizontal. On this assumption, a disk of the relative dimensions stated above would fall at the same rate as a sphere with a diameter only two-thirds as great. However, this assumption also is incorrect, as common observation of flakes of mica falling through water and of pieces of paper falling through air indicates that the average inclination is much flatter than 60° and that the clay particles probably fall with an average inclination much nearer 0° (the first assumption) than 60° (the second one).

⁴⁵ Fletcher, C. C., and Bryan, H., Modification of the method of mechanical soil analysis: U. S. Dept. Agr. Bur. Soils Bull. 84, p. 13, 1912.

Therefore it seems probable that the settling velocity of an average clay particle from these samples of shale is the same as that of a sphere with diameter much less than two-thirds but somewhat more than one-third the maximum diameter of the clay particle. However, not all the clay particles have this average shape; some are more than 15 times as long as they are thick, and others are nearly equidimensional, so that the average relationship derived here can not be used to compute the diameters from the settling velocities.

In this discussion of the effect of shape, it has been assumed that all particles have the same density. If adsorbed films cover all the surfaces the effective density of the fine and coarse grains may not be the same, and the settling velocities would bear an even more complex relation to the diameters. Evidence that the fine grains in these samples may have a greater density than the coarse grains is given on page 33.

Because of the observed differences in shape and the possible differences in density, it is clearly impossible to calculate from the settling velocities the exact diameters of the clay particles in these samples. In fact it has been recommended⁴⁶ that the settling velocities instead of the assumed diameters be generally used in mechanical analyses. However, a statement or graph of the percentages of particles with different settling velocities does not give a very clear or easily grasped picture of the size composition of a rock, and if some of the particles are large enough to be readily measured by sieving their settling velocities are difficult to determine. Odén⁴⁷ sought to give a readily understood meaning to the different settling velocities by converting them into the "equivalent" radii of spheres having the same settling velocities.

In an attempt to avoid the uncertainty and confusion that inevitably accompanies any conversion of settling velocities into exact diameters and yet at the same time to give a readily understood interpretation of the size composition of these rock samples, the writer proposes to make a short cut from the observed settling velocities to the common names "sand," "silt," and "clay." This short cut can be made by taking advantage of the differing relations that have been reported between diameters and settling velocities and the differing concepts that have been held of just what diameters are meant by these names and more or less arbitrarily defining these names in terms of the settling velocities.

⁴⁶ Robinson, G. W., On certain regularities in the mechanical composition of soils and other granular substances: IVth Internat. Conf. Pédologie [Rome] Actes, vol. 2, pp. 180-187, 1926; The grouping of fractions in mechanical analysis; First Internat. Cong. Soil Sci. (Washington), Commissions I and II, Abstracts of Proc., pp. 1-2, 1927.

⁴⁷ Odén, Sven, The size distribution of particles in soils and the experimental methods of obtaining them: Soil Sci., vol. 19, p. 3, 1925.

If the reported settling velocities and diameters of particles are plotted on double logarithmic paper and if the size limits of the names "fine sand," "very fine sand," "coarse silt," "medium silt," "fine silt," "very fine silt," "coarse clay," and "medium clay," as defined by Atterberg, Wentworth, and Udden, are plotted to scale on the same graph, it becomes apparent that a straight line based on average settling velocity and size limit can be drawn that does not differ greatly from any of the data or size classifications. The settling velocities that delimit the different size groups on this average curve are approximately as follows:

	Settling velocity (microns per second)
Very fine sand-----more than	3.840
Coarse silt-----	960-3.840
Medium silt-----	240-960
Fine silt-----	60-240
Very fine silt-----	15-60
Coarse clay-----	3.75-15
Medium clay-----	0.9375-3.75
Fine clay-----less than	0.9375

The limiting velocities between the successive size fractions decrease by the constant ratio 1 to 4. This ratio follows from the logarithmic scale of limiting diameters chosen by Atterberg, Udden, and Wentworth, and from the Stokes law (which is reliable through this range of sizes; see pp. 17-18) that the settling velocity varies as the square of the diameter.

If these somewhat arbitrary names are used for the particles with certain settling velocities, the data on the graphs showing the successive weights and the derived cumulative curves can be replotted into a more readily understood graph. On this second graph can be shown cumulative curves and pyramidal diagrams (see p. 22), also settling velocities and common names of the different fractions. (See pls. 2 and 3.) It requires slightly more than one hour to plot the readings, make the several corrections, and redraw each of these final graphs.

Accuracy of final graphs.—Duplicate and triplicate series of weighings indicate, as was to be expected from the degree of accuracy of the balance used, that for the most part the graphs are accurate within less than 2 per cent. The material represented by the part of the curve showing the most rapid velocities was tested further by sieving. After the series of weighings had been completed, several of the suspensions were washed under water through a wire sieve having square openings with a maximum or diagonal diameter of 0.09 millimeter (200-mesh). When dried and weighed, the grains of fine sand retained on this sieve were found to form only a very small proportion of every sample—a qualitative but, in view of the limits of accuracy of the measurements, not an exactly quantitative verification of the first part of

the settling-velocity curves. Judged both by the repeated weighings and by these checks afforded by sieving, the curves seem to be accurate within 2 per cent. However, the effect of other possible variations that were not tested, such as the accidental choice of an unrepresentative sample and differences in degree of disintegration, may increase the limit of accuracy to 5 per cent or more.

The most uncertain part of the graphs is the weight of the final clay fraction. The final total weight of accumulated sediment on the pan might be determined in any one of three ways:

1. Weighing may be continued until all of the sediment has settled out. This complete settling may require days or weeks or even years, the time depending upon the size of the particles and the settling distance. If the settling time is long, errors creep in because of variations in temperature, which produce convection currents and changes in viscosity (see pp. 18, 20) and because of contamination with dust. Furthermore, prolonged weighing in the water-saturated atmosphere around a settling beaker is harmful to a delicate balance. Therefore it is desirable to use the shortest possible settling time that is consistent with reasonably accurate results.

2. It seems that, if the total weight of suspended sediment and the proportion of the total liquid that lay above the weighing pan were known the weight of finer particles still remaining in suspension after the coarser part had settled could be calculated by difference. It is true that a certain proportion of the rock might dissolve in the liquid and so would never settle out; but in view of the probability that the finer particles dissolve more rapidly than the larger ones and that much of the soluble material was probably adsorbed on the finer particles, it seems more or less justifiable to include whatever proportion of material has dissolved as a part of the finer fractions.⁴⁹ On the other hand, it has been noted that the amount of sediment that actually accumulates upon the pan is less than the amount that would be expected.⁵⁰ (See p. 29.) If this loss is not due simply to the solution of a portion of the original sample, the weight of the final clay fraction can not be accurately determined by difference but must be estimated by some other method. This loss in weighing is discussed more fully under the next heading.

3. An examination of the series of successive weights that extended over longer periods of time suggests very strongly that, in these particular

⁴⁹ Fletcher, C. C., and Bryan, H., *op. cit.*, p. 13.

⁵⁰ Coutts, J. R. H., and Crowther, E. M., A source of error in the mechanical analysis of sediments by continuous weighing: *Faraday Soc. Trans.*, vol. 21, pp. 374-380, 1926. Shaw, C. F., and Winterer, E. V., A fundamental error in mechanical analysis of soils by the sedimentation method: *First Internat. Cong. Soil Sci.* (Washington), Commissions I and II, *Abstracts of Proc.*, pp. 5-9, 1927.

samples of shale, the final clay fraction can be estimated, as accurately as is warranted by the composition of the rocks and the purpose of the analysis, from a relatively short series of successive weights. This method is discussed more fully on page 30, but it may be stated here that in the writer's opinion the final weight can be estimated by this method within 5 per cent, or in any event almost certainly within less than 10 per cent of the actual weight.

Loss of sediment by the method of continuous weighing.—From the known weight and specific gravity of sediment dispersed evenly through a suspension of known volume, and from the known area and depth of a settling pan in this suspension, it seems that it might be possible to calculate the weight in water of the sediment that would eventually accumulate on the pan. However, it has been found that the amount that actually falls on the pan is usually only 65 to 95 per cent of the calculated amount. It has been suggested that this loss may be caused either by currents that deflect sideways the downward fall of particles originally above the pan⁵¹ or by a mutual repulsion among the dispersed particles that forces them toward the walls of the settling beaker.⁵²

Other possible explanations of this loss may be worth mentioning. (1) After the original weighing and during the process of disintegrating the sample, some of the sediment may be lost by adhering to the sides or stopper of the flask. (2) If the preliminary stirring is made with a rotary motion, some of the larger grains are driven centrifugally toward the walls of the settling beaker. (3) In the few seconds between the time the suspension is stirred and the time the pan is introduced into the suspension, some of the larger grains may fall below the level of the pan. (4) Some of the organic matter, moisture, alkalis, and fine particles in the original sample may become dissolved in the suspending liquid. (5) Sediment deposited near the edge of the pan may slump off, particularly if the layer of accumulated sediment becomes thick. (6) The final weighing may be made too soon, while much fine material still remains in suspension. (7) In calculating the expected weight in water of sediment, a specific gravity higher than the true one may be used inadvertently.

Most but not necessarily all of these possible sources of error would be negligible in the experiments made by Coutts and Crowther and by Shaw and Winterer. Coutts and Crowther's data indicate that the losses of silt, fine silt, and clay are about equal where the pan is appreciably smaller in diameter than the settling vessel and is more than 1 or 2 centimeters above the bottom of the vessel. These data in themselves seem

to show that neither currents nor mutual repulsion are adequate explanations of the loss, because both should affect the smallest particles more than the largest. Some of the other possible explanations, which also would call for a greater loss of one size than another, are likewise inadequate.

The apparent losses in the group of shale samples studied by the writer can be estimated fairly closely. From the weight in air of the air-dried samples, the weight in water of the accumulated sediment when it seemed that all or nearly all of the suspended particles had settled, and the grain density of each particular sample, it was determined that the apparent loss ranged from 5 to 20 per cent and averaged about 10 per cent. However, the air-dried samples contained a few per cent of moisture and organic matter. The interstitial moisture would certainly not settle out of the suspension, and the discoloration of the water in which the rock was disintegrated indicates that some of the organic matter was dissolved. In fact, these percentages of apparent loss, calculated as above indicated, increased with the percentage of water and organic matter in the air-dried samples. On assuming, then, that all the contained moisture and organic matter became a part of the suspending medium and did not settle onto the pan, and correcting the specific gravity of the particles accordingly, it was found that the losses ranged from 1½ to 10 per cent and averaged about 5 per cent; that is, the calculated losses are greatly reduced but not eliminated by correcting the determination for the moisture and organic matter in the sample.

The losses over and above those caused by moisture and organic matter might have been due to any of the causes mentioned above, but, considering the surprising thicknesses of sediment that accumulated on the weighing pan (p. 26), slumping of sediment off the edges of the pan could of itself easily account for all the losses observed.

Whatever the correct explanation, the actual losses are small; and in view of the possibility or even probability that they should be included as a part of the finer fractions (see p. 28), it seems that in these samples the weight of the final clay fraction might after all be determined by difference. However, it may seem safer, and it certainly is easier, to estimate directly the final weight in water of the sediment deposited on the pan. The final weight necessary for determination by difference depends upon the weight in air, the moisture and organic content, and the grain density; hence the calculation is laborious, but this final weight can be estimated much more easily and with sufficient accuracy, as explained in the following paragraphs.

Estimation of the final weight: exponential form of "accumulation curves."—The method used here of

⁵¹ Coutts, J. R. H., and Crowther, E. M., op. cit.

⁵² Shaw, C. F., and Winterer, E. V., op. cit.

directly estimating the final weight depends upon the fact that the "accumulation curves" of the samples studied plot as straight lines on semilogarithmic paper. After some irregularities for the first few minutes, the weight of sediment was found to increase as the logarithm of the time. Obviously this rate could not continue indefinitely, but in every

the limit of accuracy of the rest of the mechanical analysis.

This exponential form of the "accumulation curves" means that there is a certain regularity of sequence in the proportions of the material of different settling velocities. More specifically and in geologic terms, it means that below a certain size (which

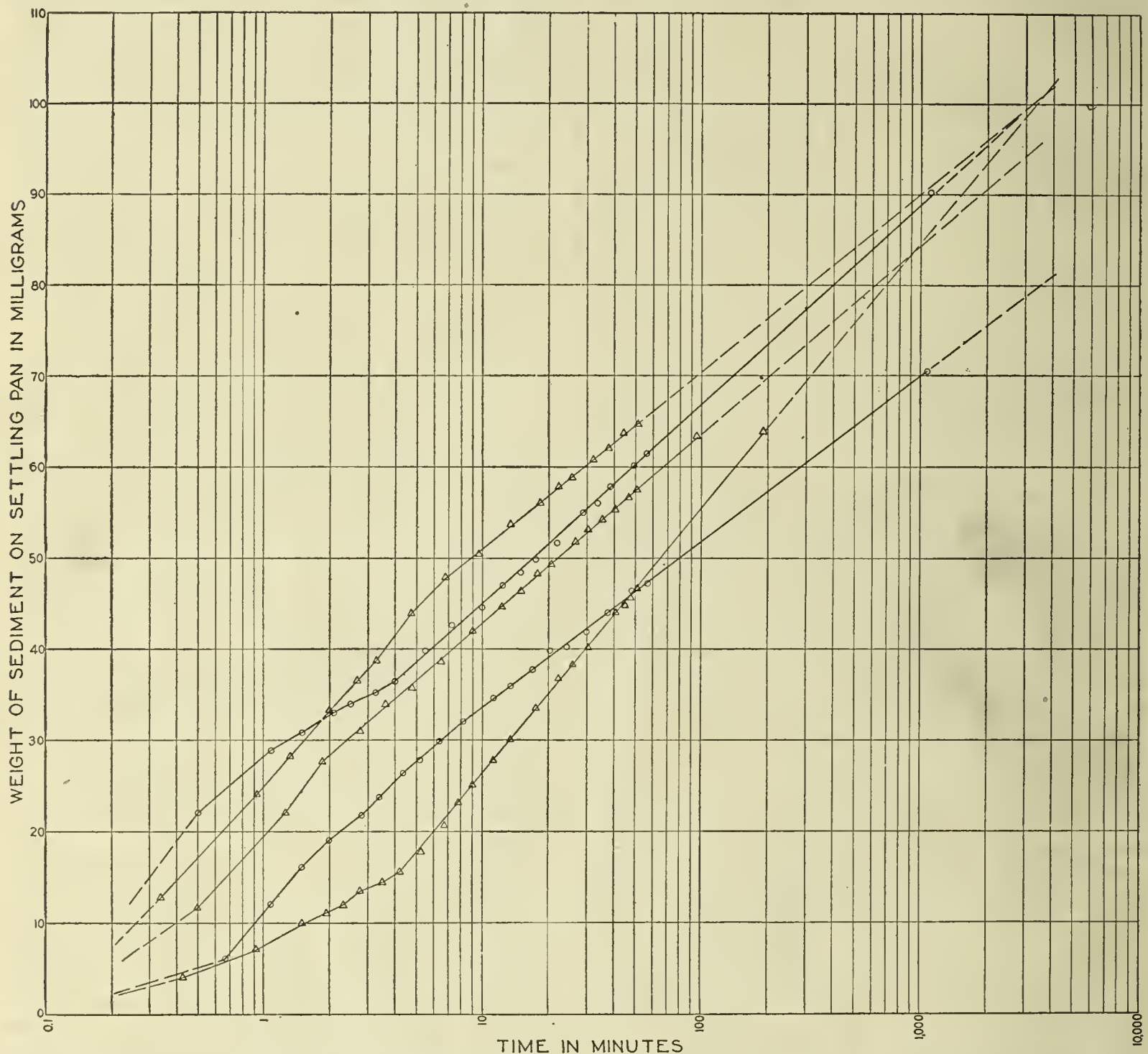


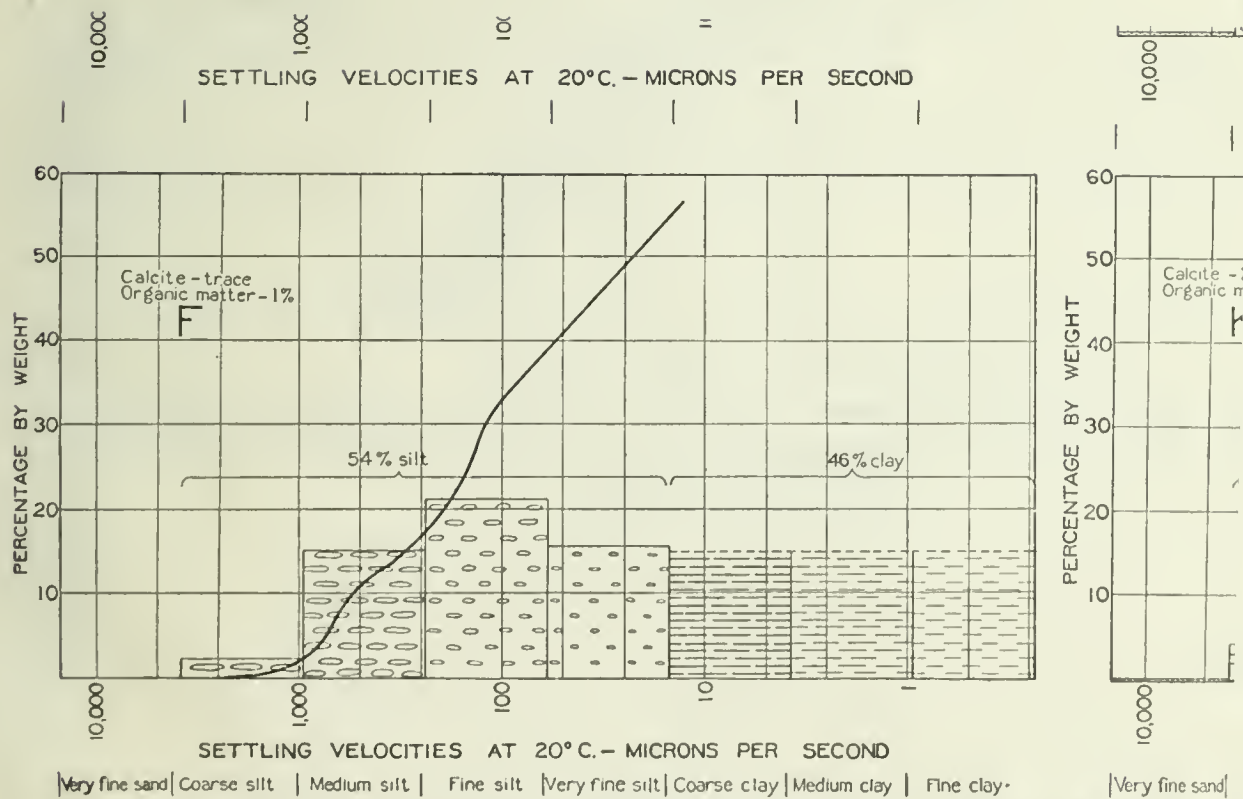
FIGURE 1.—Linear relationship between the weight of sediment on the settling pan and the logarithm of the time of settling. A graph of the actual observations made in the mechanical analysis of five rock samples from the Black Hills region. The exponential form of the "accumulation curves" here illustrated may be used to estimate the final weight of sediment

sample studied it held for as long as weighing was continued. (See fig. 1.)

The slopes of the different curves or the constants that relate the two variables in the different samples are not the same; but if the slope of the curve and the settling velocity of the finest clay particles are known, the final weight can be estimated well within

coincides roughly with the lower size limit of very fine sand) there are about equal weights of the different size fractions—that is, that the shale and mudstone are essentially unsorted. (See pls. 2 and 3.)

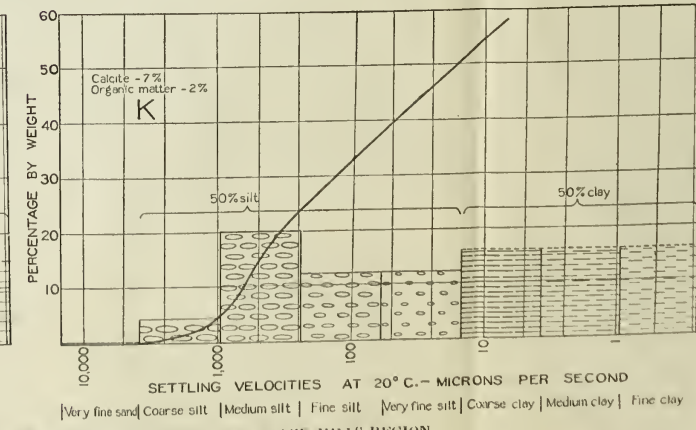
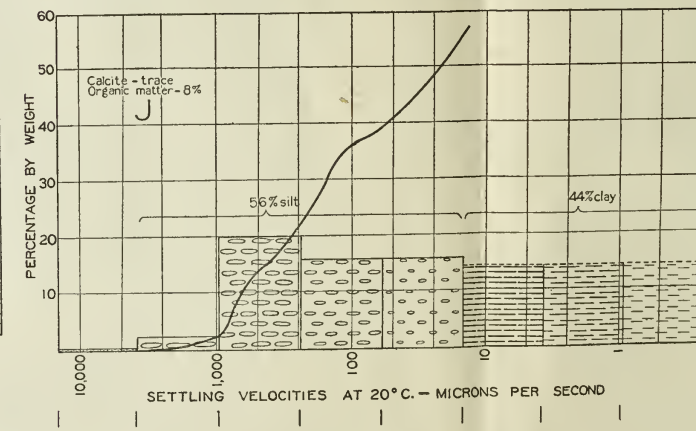
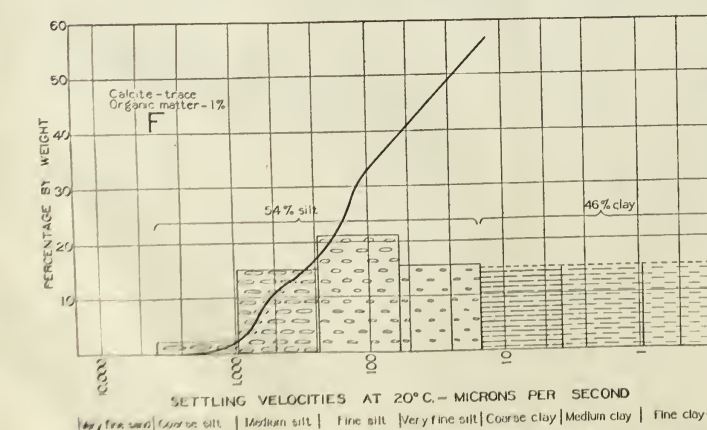
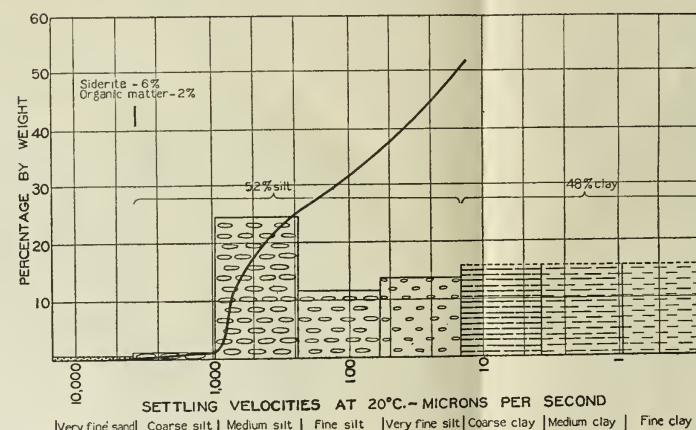
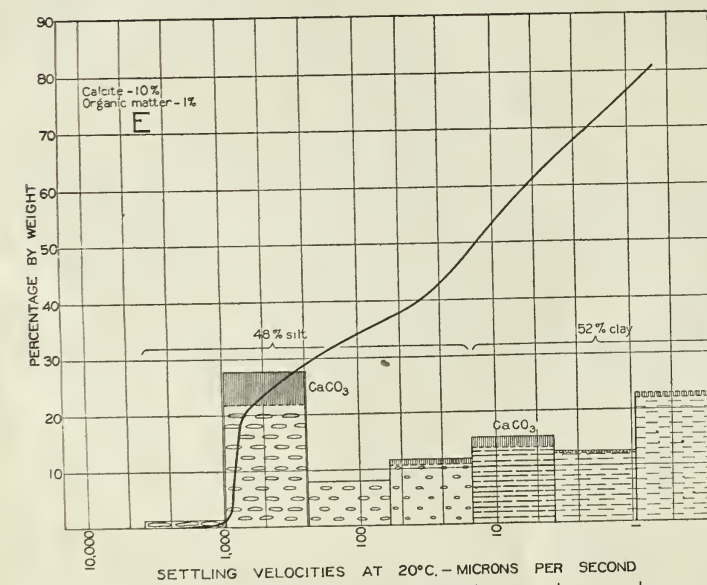
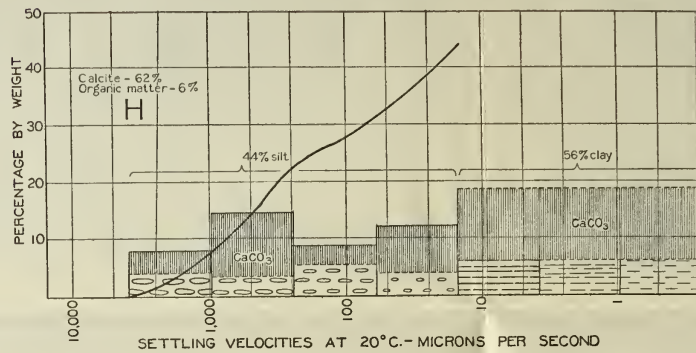
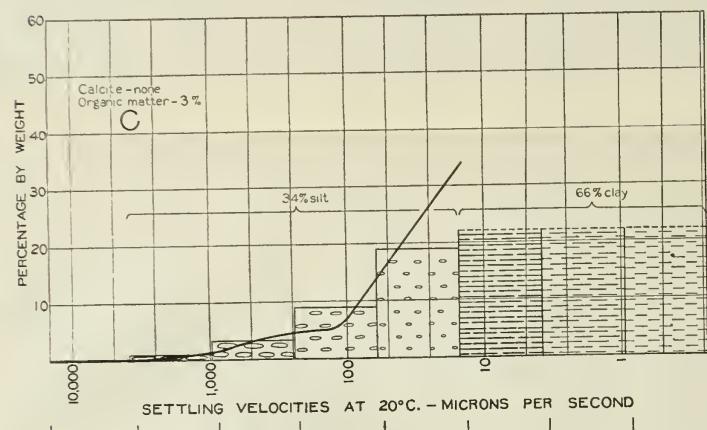
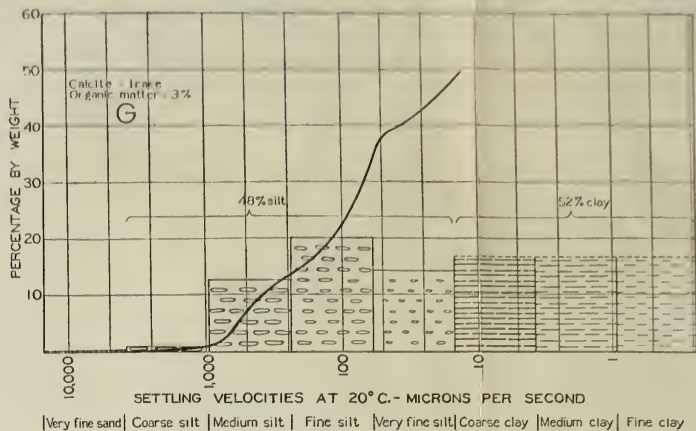
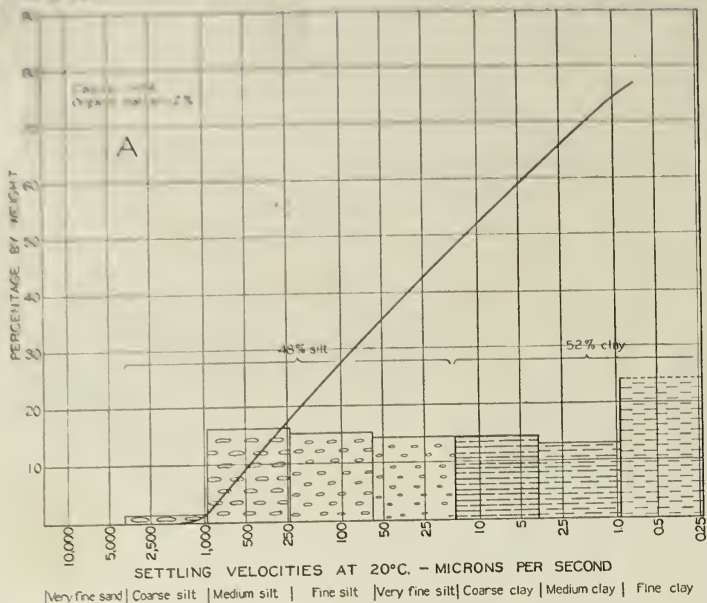
This almost complete lack of sorting means either that after deposition the particles grew into clay minerals of many different sizes (see p. 14), or that



MECHANICAL ANALYSES OF UPPER CRETACEOUS SEDIMENTARY ROCKS

Pyramidal diagrams and cumulative curves of sedimentation

- | | |
|---|---|
| A. Black shale from Skull Creek member of Graneros shale. | II. Calcareous marl from Greenhorn formation. |
| C. Black shale from Belle Fourche member of Graneros shale. | I. Gray mudstone. |
| E. Calcareous marl from Greenhorn formation. | J. Black shale. |
| F. Gray shale from upper member (Turner sandy member) of Carlile shale. | K. Gray mudstone. |
| G. Gray shale from lower member (Sage Breaks shale member) of Niobrara formation. | |

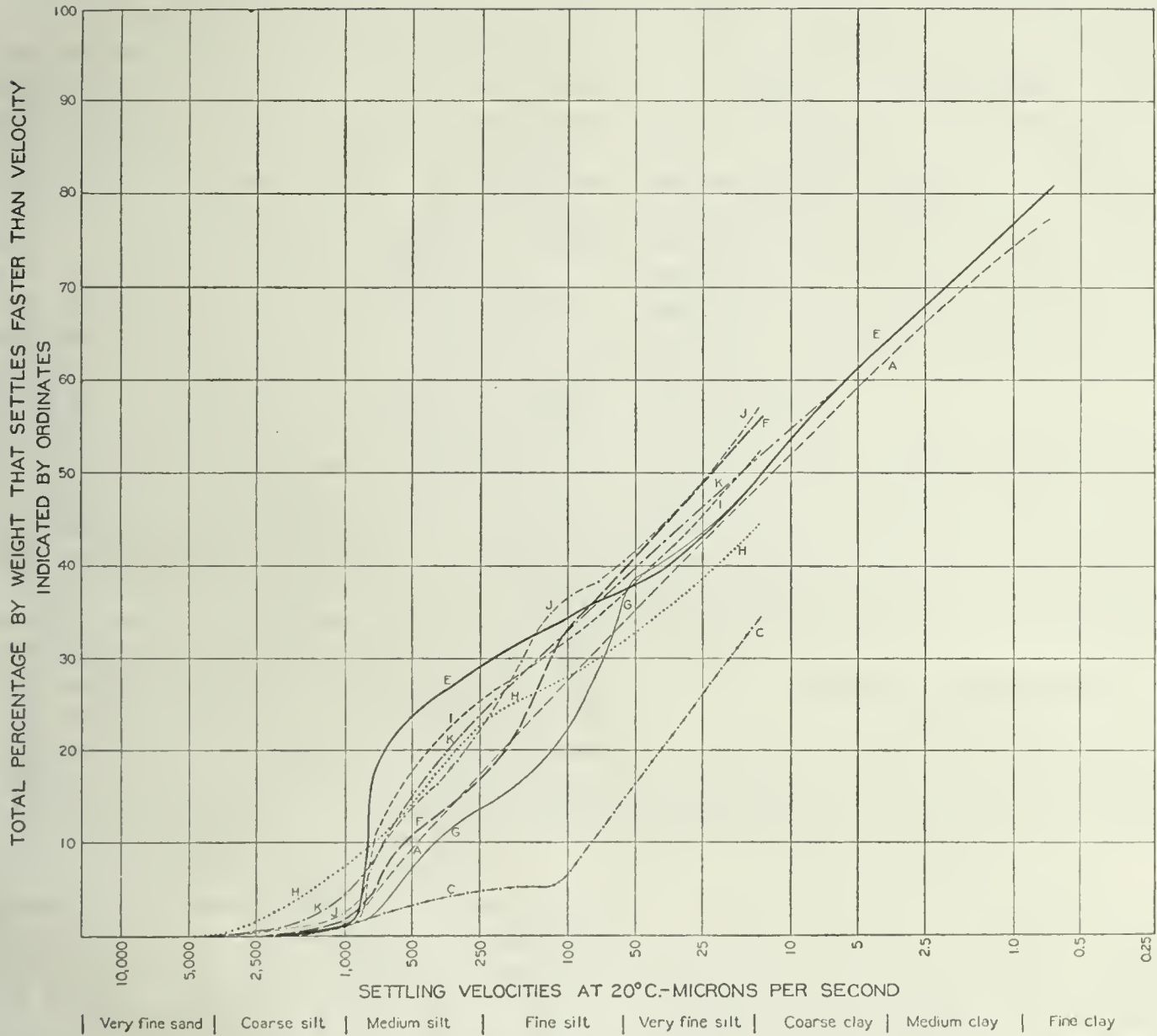


MECHANICAL ANALYSES OF UPPER CRETACEOUS SEDIMENTARY ROCKS OF THE BLACK HILLS REGION

Pyramidal diagrams and cumulative curves of each sample.

- A. Black shale from Kell Creek member of Ottumwa shale.
- C. Black shale from Belle Fourche member of Ottumwa shale.
- F. Carbonaceous mud from Fremont formation.
- E. Gray shale from upper member (Fremont formation) of Pierre shale.
- G. Gray shale from lower member (Buge Buge shale member) of Niobrara formation.

- H. Carbonaceous mud from upper member (Beaver Creek cherty member) of Niobrara formation.
- I. Gray micaceous from lower part (Champion ferruginous member) of Pierre shale.
- J. Black shale from middle part (Alloué black shale member) of Pierre shale.
- K. Gray micaceous from upper part of Pierre shale.



COMPARISON OF CUMULATIVE CURVES OF SPECIMENS OF UPPER CRETACEOUS SHALES AND MARLS FROM THE BLACK HILLS REGION

See Plate 2 for description of samples.

the detritus transported to the site of deposition and laid down there consisted of about equal amounts of silt and clay. An unsorted supply of the detritus transported to the site of deposition and a lack of sorting of the detritus during deposition might be accounted for in either of two ways: (1) Wave action might distribute unsorted mud from rivers widely over a shallow sea, and in waters that were very quiet or only gently agitated by even the most severe storms the suspended mud might settle to form an unsorted sediment or a sediment composed of alternating coarse and fine layers so thin that a small sample would appear unsorted. (2) Mud particles of many different sizes, grouped together into aggregates by flocculation in salt water, might be transported and deposited as separate aggregates, so that only those particles too large to be affected by flocculation (sand grains) would show any evidence of sorting.

Thus the lack of sorting in these rocks might be accounted for by secondary growth of clay minerals, by deposition in waters deeper than wave base, or by flocculation of different-sized particles into aggregates. It is not possible to say with any degree of assurance which of these three explanations is the most applicable to the material studied. However, the tendency toward uniform orientation and the indefinite outlines of the clay crystals (pp. 5-6), on the one hand, and the fact that the samples representing material deposited in relatively shallow water (pp. 13, 51-52, 53) are not strikingly better sorted than those representing material deposited in deeper water, on the other, suggest that neither secondary growth nor deposition below wave base adequately explains the lack of sorting. If, by elimination, flocculation is decided to be the chief factor, the possibility suggests itself that saline and fresh-water clays and shales may be distinguished by their degree of sorting.

Whatever its explanation, the lack of sorting means that the rocks consist of nearly equal amounts of silt and clay. With the single exception of sample C, which contains about twice as much clay as silt, the samples are nearly identical in their size composition, and they might with equal justification be called either "silty claystones" or "clayey siltstones."⁵³ For rocks as poorly sorted as these, the more inclusive terms "shale" or "mudstone" (depending upon whether or not the rock shows marked fissility⁵⁴) are probably preferable.

Size of particles of calcium carbonate in calcareous marls.—In general, the more calcareous samples showed a slightly higher degree of sorting than the

⁵³ Wentworth, C. K., A scale of grade and class terms for clastic sediments: Jour. Geology, vol. 30, pp. 387, 390, 1922. Twenhofel, W. H., Treatise on sedimentation, p. 186, 1926.

⁵⁴ Geikie, Archibald, Textbook of geology, vol. 1, pp. 169-170, London, 1903. Harker, A., Petrology for students, p. 216, 1919. See also footnotes on p. 39 of this report.

less calcareous samples. (See pl. 3.) Microscopic examination of thin sections suggested that nearly all the particles of carbonate in the marls are either of about the same diameter or of two very different diameters. It therefore seemed possible that the more perfect sorting of the calcareous samples might be caused by the abundance of carbonate grains in certain size fractions. Accordingly mechanical analyses were made of the insoluble residues of the more calcareous samples (E and H). From the known carbonate content and the mechanical analysis of the entire sample, the size distribution of the carbonate grains could then be worked out. Although the degree of disintegration of these insoluble residues was less satisfactory than that of the other samples, this comparison showed that most of the sorting of the calcareous specimens is in fact caused by the concentration of the carbonate in those size fractions that contain the greatest weight of particles; that is, the insoluble residues are essentially unsorted, but the carbonate grains tend to occur in definite sizes. (See pl. 2, E and H.)

This sorting of the carbonate grains may mean that, unlike the conditions attending the formation of the noncalcareous silt and clay, either the supply of carbonate grains was itself sorted, or that, after deposition, certain sizes grew at the expense of others, or that the carbonate grains were not flocculated but were left separate and therefore were sorted by the currents.

DENSITY AND POROSITY

DETERMINATIONS

The density and porosity of the shale samples were determined by P. G. Nutting, of the Geological Survey, who reported as follows:

SAMPLE F

Preliminary tests.—The shale works up readily in water to a mud. Even a small chip explodes violently if suddenly heated, indicating extremely minute pores (less than 0.1 micron) and considerable volatile matter. It readily dissolves in hydrofluoric acid, leaving a dark-brown submicroscopic powder. Burning a chip half an hour in a flame turned the surface layer (0.15 millimeter deep) yellowish white and the interior black, indicating an oxidation effect and organic material nonvolatile at 850° C.

	50° C.	100° C.	200° C.	280° C.	850° C.
Loss of water on heating:					
Chips of shale—per cent—	1.65	3.38	4.42	5.05	9.65
Ground to 100 mesh—do—	1.40	2.65	3.48	4.01	7.62
Water not recovered in 48 hours:					
Chips of shale—per cent—	.1	.3	1.0	2.1	9.0
Ground to 100 mesh—do—	.0	.1	.6	1.4	7.0

The powdered shale lost a somewhat smaller percentage on heating than the chips, probably because of losses on grinding

and sifting in an atmosphere of lower humidity (35 per cent) than the average (60 per cent).

Porosity tests, taking account of organic matter and of water in various forms: Lump volumes were determined by sand displacement, Daytona beach sand, 65-100 mesh. Grain volumes were determined by displacement of distilled turpentine, pure pinene of density 0.860 at 25° C., boiling point 158° C. This liquid adsorbs on silica and alumina much less than water. It enters pores and drives out air much more rapidly and promotes a very rapid settling of fine particles. Although free water is slightly soluble in it, adsorbed water is apparently not. The changes of density and porosity with increase in temperature to which samples had been previously heated were as follows:

Temperature (° C.)	Change		
	Grain density (gram per cubic centimeter)	Lump density (gram per cubic centimeter)	Porosity (per cent)
From 27 to 120.....	+0.247	-0.084	+8.8
From 120 to 260.....	+0.072	-0.003	+1.7
From 260 to 850.....	-0.405	-0.297	+1.4

The porosity of this shale is high and dependent upon temperature. The effect of heating on the densities of lumps and of grains is considerable and significant of important internal changes.

Heating the powdered shale (finer than 100 mesh) from 27° to 120° C. increases the grain density 7 per cent, probably by removal of adsorbed water. The small change (2 per cent) from 120° to 260° is such as would be caused (in this temperature range) by dehydration of vegetable organic matter. The large decrease in density (13 per cent) between 260° and 850° C. can hardly be due to loss of material of density higher than 3. for volatile materials of such high density do not exist. The alternative conclusion is that the grains themselves lose internal water or other molecules through pores so minute that the pycnometer liquid (pinene, C₁₀H₁₆) can not enter them.

Heating a lump of this shale lowers its density by driving off interstitial and confined volatile matter (water?), thus decreasing its mass without appreciable change in volume. The change is 4.2 per cent between room temperature (27° C.) and 120° C., practically nil between 120° and 260° C., and 15 per cent between 260° and 850° C. The smaller change at low temperatures is probably due to loss of pore and adsorbed water. The larger change at high temperatures is probably caused by loss of combined water from hydrous aluminosilicates and organic matter. On the other hand, considerable dehydrated but oxidizable organic matter is left even at 850° C., as is shown by the dark-gray interior and by the residue on solution in hydrofluoric acid.

That is to say, as the shale is heated above room temperature its porosity increases, rapidly at first and then more and more slowly. This increase of porosity is caused partly by a slight decrease in lump density and partly by a large initial increase in grain density. Up to 260° these two changes are such that the lump density of the water-saturated specimen $\left(\text{lump density plus } \frac{\text{porosity}}{100}\right)$ is virtually constant. This indicates that the changes in grain and lump densities below 260° are due almost entirely to loss of water, which has a density of 1.0. Furthermore,

the rock loses weight upon heating, but a variable part of this loss is recovered in 48 hours, and this recoverable part of the loss reaches a maximum at a temperature of about 200°. These two sets of observations indicate that the change in porosity below 200° is almost entirely caused by loss of interstitial and adsorbed water, constituents that are not an essential part of the mineral grains in the shale. Therefore 200° C. seemed to be the most satisfactory temperature at which to heat the entire group of samples before determining their porosity. The report on the full series of samples follows:

All samples were given an oven treatment of 200° for at least 16 hours to remove pore water and part of the adsorbed water, but leaving combined water to be included with grain material. Grain density was determined on material crushed to pass a 100-mesh sieve (0.15 millimeter opening), and reheated to 200° C. for at least 2 hours. Blind pores in the 100-mesh lumps are believed to be negligible when pinene is used as pycnometer fluid.

Sample	Grain density	Lump density	Pore space (per cent)
K.....	2.628	1.961	25.4
J.....	2.428	1.559	35.8
I.....	2.755	2.038	26.0
H.....	2.640	1.970	25.4
G.....	2.670	1.995	25.3
F.....	2.624	1.999	23.8
E.....	2.795	1.743	37.6
C.....	2.664	1.776	33.3
A.....	2.765	1.866	32.5
Average.....	2.66	1.88	29.5

The density and porosity of samples B (hard siliceous shale from the Mowry member of the Graneros shale) and D (pyritic limestone from the Greenhorn formation) were not determined.

GRAIN DENSITY

The grain density or specific gravity of the mineral fragments in the samples ranges from 2.428 to 2.795 and averages about 2.66. Comparison with the chemical analyses shows that the density depends upon the amount and kind of impurities in the rock. The sample with the lowest grain density (sample J) is the one that contains the most organic matter; the one with the highest density (sample E) is one of those that contain most calcite; and another sample with high grain density (sample I) contains much siderite.

From the chemical analyses it is possible to work backward and estimate the grain density or specific gravity of the clayey material. After taking out the proportions of moisture,⁵⁵ organic matter,⁵⁶ carbon-

⁵⁵ The moisture was determined at 105°, but the grain density at 200°. However, the resulting error in the computations is negligible. If the loss of water on heating from 105° to 200° is similar to that in sample F, the computed grain densities are too low by only a few points in the third decimal place.

⁵⁶ Density of organic matter taken as 1.5.

ates, and pyrite indicated by the chemical analyses (see p. 8), the density of the remainder of the rock can be computed. With these corrections, the computed grain density of the remaining clayey material ranges from 2.57 to 2.88. Not all these calculations are equally trustworthy, for in some of the samples the proportion of carbonates and organic matter is large, and in others it is small. If the relative validity of the different calculations is taken into account the weighted average grain density of samples that have been heated to 200° C. is about 2.72. If the grain density increases with temperature as in sample F, this average would correspond with a density of about 2.45 if the samples had not been heated above room temperature.

The corrected grain densities are not exactly (perhaps not even approximately) those of a pure clay mineral. Microscopic examination shows that some samples contain noticeable proportions of quartz sand or silt, and it is interesting to note that these sandy samples have lower density than the others. The mechanical analyses also indicate that the larger the proportion of coarse grains the lower the density.

Apparent relation between corrected grain density of the clayey material (total rock minus moisture, organic matter, carbonates, and pyrite) and percentage by weight of silt (grains coarser than clay size)

Sample	Density	Silt (per cent)
J.....	2.57	56
F.....	2.65	54
K.....	2.66	50
C.....	2.72	34
G.....	2.73	48
I.....	2.75	52
A.....	2.82	48
E.....	2.85	48
H.....	2.88	44
Weighted average.....	2.72	

This apparent inverse relation between the size and density of the grains is somewhat surprising, but it might be explained in any one of several ways. The determinations of grain density might involve a systematic error. For example, more inclusions of gas and liquid may be left in large particles than in small ones.⁵⁷ Or compression of the liquid film around small particles may cause the observed value of density to be greater than the true density.⁵⁸ However, neither of these possible explanations seems adequate to account for the apparent relation between size and density, and some other explanation is necessary. The clayey material (the total rock minus moisture, or-

ganic matter, carbonates, and pyrite) may consist chiefly of two ingredients—one coarse and light (perhaps quartz), the other fine and heavy⁵⁹ (possibly a clay mineral). Increasing the proportion of the fine heavy ingredient would thus give the effect noted. Or, on the other hand, the clayey material may consist of one chief ingredient, the particles of which are coated with a film of some heavier material. The finer the particle the greater its relative surface and hence the greater might be its average density. The correct explanation is not known.

A relation between the proportion of combined water and these corrected grain densities might be expected, but no such relation could be discovered.

LUMP DENSITY

The lump or rock density ranges from 1.559 to 2.038 and averages about 1.88. If the lump and grain densities vary with the temperature to which the samples have been heated, as in sample F, this average corresponds to room-temperature lump densities of about 1.96 (dry) and 2.1 (saturated with water). Corrected for the dip of the beds where sampled (as explained on pp. 35-36, the average room-temperature lump densities are about 1.7 (dry) and 2.0 (wet).

Data on the lump density of sedimentary rocks are greatly needed for estimating the weight of overburden at different depths below the surface and for interpreting the gravity anomalies, or differences between the observed gravity and that calculated by the assumption of isostatic compensation at a certain depth.⁶⁰ Particularly are these data needed near the Black Hills, as this region is one of exceptionally large and widespread positive anomalies.⁶¹ These anomalies might conceivably have been caused by rocks heavier than the average near the surface, but these determinations show that the Upper Cretaceous shales are not heavier than the average; in fact, even without making corrections for the squeezing that some of the samples have undergone in folding (see pp. 35-38), they are unusually light. Therefore, as more than 4,000 feet of these light fine-grained sedimentary rocks underlie the gravity station at Moorcroft, Wyo., the actual positive anomaly there is even greater than the computed one. These determinations thus indicate that the basement rocks are exceptionally

⁵⁹ It has been found that, among other differences in composition, the finer-sized portions of soils and clays commonly contain more of the heavy iron oxides than the portions of intermediate size. Robinson, W. O., and Holmes, R. S., The chemical composition of soil colloids: U. S. Dept. Agr. Bull. 1311, 1924. Grout, F. F., The relation of texture and composition of clays: Geol. Soc. America Bull., vol. 36, pp. 402-403, 414-415, 1925.

⁶⁰ White, David, Gravity observations from the standpoint of the local geology: Geol. Soc. America Bull., vol. 35, pp. 209-218, 1924.

⁶¹ Idem, pp. 250-260, 275. Bowie, William, Isostatic investigations and data for gravity stations in the United States established since 1915: U. S. Coast and Geodetic Survey Special Pub. 99, fig. 7, 1924; Isostatic condition of the United States as indicated by groups of gravity stations: U. S. Coast and Geodetic Survey Serial 366, pp. 6-7, illus. opp. p. 2, 1926.

⁵⁷ Sosman, R. B., The properties of silica, pp. 299-300, 304, Chem. Catalog Co., 1927.

⁵⁸ Williams, A. M., Two properties of powders: Faraday Soc. Trans., vol. 18, pp. 87-90 (especially p. 88), 1922. Harkins, W. D., and Ewing, D. T., A high pressure due to adsorption and the density and volume relations of charcoal: Am. Chem. Soc. Jour., vol. 43, pp. 1787-1802, 1921.

heavy or unusually near the surface in the Black Hills region, or else that the crust is overloaded there.

Sorby, Hedberg, and the writer have called attention to the fact that the porosity of argillaceous rocks commonly decreases (and hence the lump density increases) with increasing pressure or depth. The empirical relation pointed out by Sorby⁶² and by Hedberg and the writer⁶³ can be generalized into the statement that the lump density increases as $\frac{GD+a}{D+b}$, in which G is grain density, D is depth below the present surface, and a and b are constants that depend upon the thickness of rocks eroded from above the present surface, the initial porosity of the rock, the grain density, and the extent to which the pores are filled with water.

Inasmuch as the load of the overburden increases as the integral of lump density to depth,⁶⁴ this relation means that the overburden increases as

$$GD - (Gb - a) \log \frac{D+b}{b}.$$

Argillaceous rocks are very abundant at the earth's surface, and empirical relations such as that above set forth, if they can be verified, will be useful as a means of estimating the distribution of density in the upper few miles of the earth's crust.⁶⁵ However, the lump density of a rock depends upon both the grain density and the porosity, and if the grain densities in a series of samples are not constant these possible relations can be tested most simply by an examination of the porosity.

POROSITY

The porosity, or percentage by volume of pore space, in these samples previously heated to 200° C. ranges from 23.8 to 37.6 and averages 29.5. If the porosity varies with the temperature to which the sample has been heated, as in sample F, this average corresponds to a porosity of about 20 per cent at room temperature. This large difference is due almost entirely to interstitial and adsorbed water in the shale sample (p. 32) at room temperature, and the porosity of a sample that has been heated to 200° gives a much more accurate picture of the volume composition of the rock. Corrected for the dip of the beds where sampled (see pp. 35-36), the average porosity at room temperature of the rocks before tilting is about 30 per cent.

⁶² Sorby, H. C., On the application of quantitative methods to the study of the structure and history of rocks: *Geol. Soc. London Quart. Jour.*, vol. 64, pp. 227-231, 1908.

⁶³ Hedberg, H. D., The effect of gravitational compaction on the structure of sedimentary rocks: *Am. Assoc. Petroleum Geologists Bull.*, vol. 10, pp. 1057-1058, 1926. Rubey, W. W., The effect of gravitational compaction on the structure of sedimentary rocks—a discussion: *Idem.*, vol. 11, pp. 621-632, 1334, 1927.

⁶⁴ Nutting, P. G., The deformation of granular solids: *Washington Acad. Sci. Jour.*, vol. 18, pp. 123-126, 1928.

⁶⁵ Williamson, E. D., and Adams, L. H., Density distribution in the earth: *Washington Acad. Sci. Jour.*, vol. 13, pp. 413-428, 1923.

This rather high porosity and the fact that the samples were collected from surface outcrops suggest that they may be weathered and cracked. However, this seems unlikely, for microscopic examination of thin sections cut from other fragments of the same samples disclosed neither weathering nor minute fracturing, and the fragments tested for porosity were carefully chosen. Also, Hedberg's shale samples,⁶⁶ taken from well cuttings and outcrops near wells in Kansas, have porosities that fall into a smooth curve—that is, the porosities of the outcrop samples are not disproportionately higher than those of the deeper samples.⁶⁷ This indicates that in western Kansas, at least, the porosities of the outcrop samples are as reliable as those of the deeply buried samples. Furthermore, the fact that the porosities of the samples from the Black Hills region show expectable and even quantitative relations to other variables (pp. 35-38) suggests that they are essentially correct.

In a coarse or medium-grained sandstone with 30 per cent porosity water and oil can circulate with comparative freedom, but in a shale with the same porosity the pores are much smaller, and because of surface tension and friction the liquids can scarcely move. Thus, although the shales in this region seem to have higher porosity than the oil-bearing sandstones,⁶⁸ they can still, where unfractured, act as impervious layers and confine the oil to the sandy beds. Where the shales are fractured they may serve as reservoirs for oil. The writer estimated that one-fifth of the oil produced in the Osage oil field in 1923 came from fine-grained shale beds in the Belle Fourche, Mowry, and Skull Creek members of the Graneros shale; and oil has been obtained from fractured shale beds in many oil fields in the Rocky Mountain States.⁶⁹ However, the openings from which this oil is recovered probably have no relation to the porosity of small samples of the shale.

The porosity of an argillaceous rock depends upon many factors, chief among which are the size and shape of the grains, the porosity at the time of deposition, and the amount of squeezing or compacting the rock has undergone since deposition. The presence of many small and flat grains in a rock apparently increases its original porosity. Experiments have shown that both the porosity of different samples of clay and claylike aggregates at equal pressures and the loss of the porosity of these samples with equal increments of pressure increase with increasing flat-

⁶⁶ Hedberg, H. D., *op. cit.*, p. 1052.

⁶⁷ Rubey, W. W., *op. cit.*, pp. 626-627.

⁶⁸ Collier, A. J., The Osage oil field, Weston County, Wyo.: *U. S. Geol. Survey Bull.* 736, pp. 96-98, 1922.

⁶⁹ Wegemann, C. H., The Salt Creek oil field., Wyo.: *U. S. Geol. Survey Bull.* 670, pp. 36-37, 1918. Estabrook, E. L., and Rader, C. M., History of production of Salt Creek oil field, Wyo.: *Petroleum Development and Technology in 1925*, pp. 209-211, *Am. Inst. Min. Eng.*, 1925.

ness and smallness of the constituent particles.⁷⁰ The porosity at the time of deposition probably depends upon the conditions of sedimentation, the salinity of the water, and the size, shape, and degree of sorting of the constituent particles in the mud.⁷¹ (See pp. 16, 19, 26.) After deposition, the mud is squeezed or compacted, either by folding or regional alteration⁷² or by the weight of overlying rocks.⁷³

The porosity of sandstones in this region seems to decrease with depth. A diamond-drill core at Osage, Wyo., through the Newcastle sandstone, Skull Creek shale, and Fall River or so-called Dakota sandstone shows porosity decreasing downward.⁷⁴

Of these various relations, the most obvious one shown by the porosity of the shale samples studied is the relation to the degree of deformation that the rocks have undergone. In general, the more steeply dipping rocks have the lower porosity, as indicated in the following table:

Relation of porosity to degree of deformation

Sample	Degree of deformation		Porosity (per cent)
	Dip of rocks where sampled (°)	Proximity to faults	
E	1		37.6
C	4		33.3
A	5		32.5
I	5	(a)	26.0
K	7	(a)	25.4
H	10	(a)	25.4
F	33		23.8
J	45		35.8
G	50		25.3

(a) Near faults.

This decrease of porosity with increase of dip is of considerable structural significance, for it shows that

⁷⁰ Hardy, F., The physical significance of the shrinkage coefficient of clays and soils: *Jour. Agr. Sci.*, vol. 13, pp. 243-264, 1923. Russell, J. C., and Burr, W. W., Studies on the moisture equivalent of soils: *Soil Sci.*, vol. 19, pp. 251-266, 1925. Wintermeyer, A. M., Adaptation of Atterberg plasticity tests for subgrade soils: *Public Roads*, vol. 7, pp. 119-122, 1926. Terzaghi, C., Simplified soil tests for subgrades and their physical significance: *Idem*, pp. 153 et seq. Hedberg, H. D., op. cit., pp. 1049-1050. Rubey, W. W., op. cit., p. 624. Gilboy, Glennon, The compressibility of sand-mica mixtures: *Am. Soc. Civil Eng. Proc.*, vol. 54, pp. 555-568, 1928. Rubey, W. W., The compressibility of sand-mica mixtures [discussion]: *Idem*, pp. 1936-1938. Also unpublished data furnished to the writer by E. F. Kelley and C. A. Hogenotler, of the Bureau of Public Roads, Department of Agriculture, show that a close relation exists between the porosity and total clay and silt content of soil samples.

⁷¹ Hedberg, H. D., op. cit., pp. 1039-1042. Rubey, W. W., op. cit., p. 1334. Barnes, Carl, Subsidence of fine solid particles in liquids: *U. S. Geol. Survey Bull.* 36, pp. 31, 35, 1886. Pickering, S. U., Flocculation: *Roy. Soc. London Proc.*, vol. 94A, pp. 315-325, 1918.

⁷² Hedberg, H. D., op. cit., pp. 1043, 1071-1072. Wilson, J. H., Lithologic character of shale as an index of metamorphism: *Am. Assoc. Petroleum Geologists Bull.*, vol. 10, pp. 625-633, 1926. Russell, W. L., Porosity and crushing strength as indices of regional alteration: *Idem*, pp. 939-952.

⁷³ Sorby, H. C., op. cit., pp. 227-231. Terzaghi, Charles, Principles of soil mechanics: *Eng. News-Record*, vol. 95, pp. 742-746, 796-800, 832-836, 874-878, 912-915, 987-990, 1026-1029, 1064-1068, 1925. Hedberg, H. D., op. cit., p. 1035-1072.

⁷⁴ Collier, A. J., The Osage oil field, Weston County, Wyo.: *U. S. Geol. Survey Bull.* 736, pp. 80, 97, 1922.

the shale beds were not simply tilted by folding but were squeezed and deformed internally. That is to say, they behaved as incompetent rocks and hence very probably formed similar, not concentric or parallel folds. This interpretation furnishes a theoretical basis for a quantitative statement of the decrease of porosity with increase of dip. The porosity and hence also the relative volume of the different samples vary roughly as the cosine of the angle of dip. Now if we

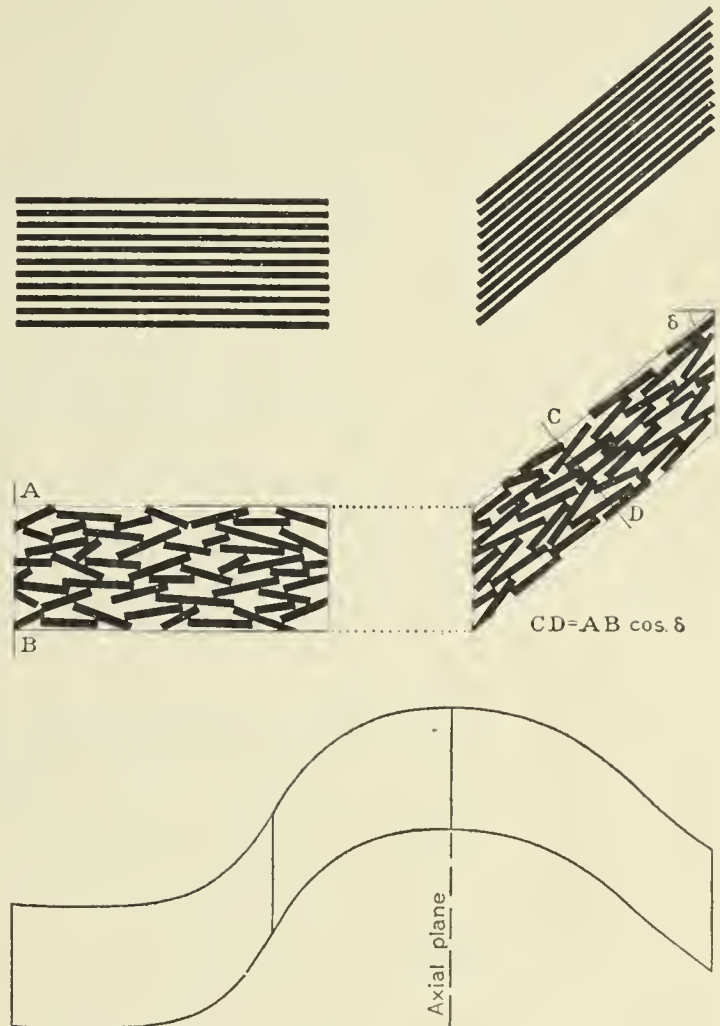


FIGURE 2.—Probable explanation of decrease of porosity with increase of dip. Whether the particles touch loosely or are held apart by cushions or envelopes of adsorbed films that cover the surface of each particle, the pores are squeezed flatter as the beds are tilted. The total volume of the pores and the stratigraphic thickness of the bed decrease, but the length of the bed and its vertical thickness remain essentially unchanged. Deformation of this type would form similar folds with vertical axial planes.

assume the simplest possible type of similar folding (horizontal shortening and vertical axial planes)⁷⁵ this is exactly the relation that we would expect to find. As the beds are tilted, the spaces or pores between the clay particles are squeezed flatter, the contained fluids being squeezed out, so that, until all pores have been closed up, the thickness of the bed decreases as the cosine of the angle of dip. (See fig. 2.)

⁷⁵ Rubey, W. W., Determination and use of thicknesses of incompetent beds in oil-field mapping and general structural studies: *Econ. Geology*, vol. 21, pp. 334-339, 1926.

Although at first thought this interpretation may seem to conflict with the principle of dilatancy⁷⁶ or the expansion of granular masses when deformed, more careful consideration shows that there is no such conflict. Granular aggregates, like clay, that consist of very small particles have a much wider range of porosity than aggregates of large spheres, perhaps because of the poor sorting, angularity, and flatness of the clay particles and the greater proportionate thickness of the deformable adsorbed layer on the particles.⁷⁷ Aggregates with porosities greater than the minimum porosity or, more accurately, aggregates with the so-called open-packing porosity are deformed plastically and without increase of volume.⁷⁸ Hence clays with porosities greater than the minimum limit, which in clays is probably only a few per cent, are not dilatant but can be deformed plastically and by actual decrease instead of increase of volume.

The rate at which the rocks are deformed and the amount of overburden at the time of deformation are probably additional factors in determining whether or not rocks are dilatant. Under light load and sudden deformation even the most plastic clay is likely to fracture and shear and so increase its volume. But it is possible that under heavy load and very slow deformation even indurated and brittle shale may adjust itself internally so that its volume is decreased instead of increased.

If the proposed interpretation is correct, the porosity of the samples before they were tilted can be determined, because the porosity after tilting depends upon the total volume of pores and mineral grains, and this total volume in turn depends upon the angle of dip and the original porosity. (See fig. 2.) Let

$$P_u = 100 \frac{V_u}{V_u + s} = \text{porosity of the untilted rock.}$$

$$P_d = 100 \frac{V_d}{V_d + s} = \text{present porosity after tilting.}$$

d = present angle of dip.

s = volume of solid particles or mineral grains in rock.

V_u = volume of voids or pores between solid particles before tilting.

$$V_d = \frac{P_d s}{100 - P_d} = \text{volume of voids after tilting.}$$

And

$$V_d + s = (V_u + s) \cos d = \text{assumed volume of rock after tilting.}$$

Then

$$V_u + s = \frac{V_d + s}{\cos d} = \text{original volume of rock.}$$

$$V_u = \frac{V_d + s}{\cos d} - s.$$

$$\begin{aligned} P_u &= 100 \frac{V_u}{V_u + s} = 100 \frac{\frac{V_d + s}{\cos d} - s}{\frac{V_d + s}{\cos d}} = 100 \frac{V_d + s - \cos d s}{V_d + s} \\ &= 100 \frac{\frac{P_d s}{100 - P_d} + s - \cos d s}{\frac{P_d s}{100 - P_d} + s} \\ &= 100 \frac{P_d + 100 - P_d - \cos d (100 - P_d)}{P_d + 100 - P_d} \\ &= 100 - \cos d (100 - P_d) \end{aligned}$$

Applying this equation shows that, as might be expected, the corrected porosities tend to decrease with depth, as indicated in the following table, and the exceptions to this regular decrease seem to be explainable as the result of other disturbing influences. Samples K, I, and H were collected near faults, and hence may be assumed to have undergone more deformation than the dip of the rocks alone would indicate. The slight departure of sample E or F from this regular decrease and other irregularities not apparent unless the porosities are plotted seem to be caused by differences in texture. (See p. 38.)

Apparent decrease of porosity with increase of stratigraphic depth

Sample	Porosity of untilted rock (present porosity corrected for dip; per cent)		Approximate stratigraphic depth below horizon of sample J (feet)
	Samples near faults	Other samples	
K-----	26+		
J-----		54.6	0
I-----	26+		
H-----	27+		
G-----		52.0	1,300
F-----		36.0	1,550
E-----		37.6	1,850
C-----		33.4	2,100
A-----		32.8	2,900

In other areas the porosity and depth of shale samples from wells are related to one another in such a way that it seems possible to estimate the thickness of rocks once present but now eroded from above the highest sample.⁷⁹ This method of estimation has not been adequately tested, and even if it is valid, it might not apply to the porosity of a few outcrop samples taken from a large area in which the thickness of overlying formations and the dip vary considerably. Nevertheless, when applied to the samples from the Black Hills region the method yields results that seem to be qualitatively correct. These results therefore tend to justify both the method of estimating maximum overburden and the interpretation of the relation between porosity and dip.

⁷⁶ Mead, W. J., The geologic rôle of dilatancy: Jour. Geology, vol. 33, pp. 685-698, 1925.

⁷⁷ Idem, p. 686.

⁷⁸ Idem, pp. 692, 697.

⁷⁹ Rubey, W. W., The effect of gravitational compaction on the structure of sedimentary rocks—a discussion: Am. Assoc. Petroleum Geologists Bull., vol. 11, pp. 625-628, 1927.

Calculated by this method from the corrected porosities and the depths below sample J, the thickness of overburden eroded from above the horizon of sample J may have been about 3,300 feet. This calculated thickness, compared with the average thickness of overlying formations that crop out farther west, is equivalent to that of all of the overlying marine Upper Cretaceous and continental Lance formations and part of the continental Fort Union formation.

However, the thickness of the formations that overlie the bed yielding sample J varies considerably from place to place, and hence the relative depths of the different samples below the higher formations are not the same as their relative depths below the horizon of sample J. The thickness of overburden should be calculated from the depths of the different samples below the highest bed once present in the area, but as the exact stratigraphic position of this highest bed is not known, the thickness must be calculated from depths below several of the higher formations. Under this procedure the method of estimating the thickness of overburden loses its false appearance of accuracy, for no two calculations give the same result. However, they all agree in indicating that the top of the maximum overburden lay somewhere between the base of the Lance and a horizon several thousand feet above the base of the Wasatch formation. That is to say, they all indicate that uplift and erosion began, or, more accurately, that sedimentation above the site of the samples ceased, sometime during the deposition of the continental Lance, Fort Union, or Wasatch formations or possibly somewhat later. These estimates are based, of course, upon the thickness of the formations as they crop out some distance west of the Black Hills uplift, and if before erosion these formations were thinner east of their present outcrops, the estimates would correspond with somewhat later epochs than those given.

Field evidence and regional relations indicate that, for the most part, the Black Hills rose after part of the Wasatch formation had accumulated; but uplift may have started much earlier and continued somewhat later than the time of maximum uplift. It is thus seen that the calculated thickness of overburden agrees with the field evidence in a general qualitative way but not with quantitative exactness.

A further test can be made both of the method of estimating the maximum overburden and of the interpretation of the relation between porosity and dip. If this method and this interpretation are both essentially correct, porosities calculated from them should differ from observed porosities in accordance with other disturbing influences such as variations in rock texture. The calculated porosities necessary for this comparison might be obtained by using those

figures for B (eroded depth) and C (the constant) in the modified Sorby depth-porosity equation⁸⁰ that best fit the data. But a more independent method, which does not demand so much pyramiding of poorly established relations and which uses only assumptions that are based upon independent evidence, is somewhat more convincing.

By starting with the assumption that the porosities decrease with depth according to the general form of the modified Sorby depth-porosity equation (an assumption based on studies made outside of the Black Hills region) and obtaining the one constant necessary for the use of this equation from field evidence that is independent of the porosity data, all porosities can be reduced to one depth.

The constant B can be eliminated algebraically.

$$(D+B)\frac{P}{100-P} = C \quad (1)$$

in which D is depth below the surface, B is thickness of rocks eroded from above the surface, P is porosity, and C is a constant. Then

$$P = \frac{100C}{B+C+D} \quad (2)$$

$$D = \frac{100C}{P} - B - C \quad (3)$$

Let P_1 =porosity at depth D_1 , and P_2 =porosity at some greater depth, $D_2=D_1+x$.

Then

$$P_2 = \frac{100C}{B+C+D_2} = \frac{100C}{B+C+D_1+x} = \frac{100C}{B+C+\left(\frac{100C}{P_1} - B - C\right) + x} = \frac{100CP_1}{100C+P_1x} \quad (4)$$

The remaining constant, C , might be obtained from data on series of porosities from other regions. The value of this constant is relatively unimportant, for throughout a wide range the precise value chosen for it does not affect the relative order of calculated porosities. However, in order to make the figures concrete, the constant can be evaluated by introducing the assumption, based on field observations, that sedimentation above the rocks represented by these samples ceased about the time that Wasatch deposition began farther west. The average of the nine values of C obtained from this assumption is approximately 2,200, and, by putting this average value into equation (4), the theoretical porosities (uncorrected for dip) that might be expected at a depth of 7,000 feet below the base of the Wasatch formation can be computed.

Then by introducing a third assumption that the stratigraphic thickness of a bed of shale decreases as

⁸⁰ See footnotes 62 and 63.

the cosine of the angle of dip (an assumption that can be more or less justified by observed variations in the thickness of folded shale beds in this region), the effect of dip on porosity can be eliminated by the

equation, $P_u = 100 - \cos d (100 - P_{7000})$. See p. 36.)

The resulting porosities, now corrected for depth and dip, show a general increase with increasing percentage of silt (that is, the coarser) grains.

Apparent relation between porosity and grain size of samples

Sample	Actual porosity of samples P_0	Approximate depth of sample below base of Wasatch formation (feet) Dw	Interval in feet of sample above bed 7,000 feet below base of Wasatch formation $x = 7,000 - Dw$	Computed porosity of rock 7,000 feet below base of Wasatch formation ^a $P_{7,000} = \frac{100CP_0}{100C + P_0x}$	Dip of rocks where sampled d	Computed porosity of untilted rock 7,000 feet below base of Wasatch formation $P_u = 100 - \cos d (100 - P_{7,000})$	Percentage of silt (grains coarser than clay size)
G	25.3	5,450	1,550	21.5	50	49.5	48
J	35.8	4,200	2,800	24.6	45	46.5	56
F	23.8	5,700	1,300	20.9	33	33.7	54
A	32.5	7,000	0	32.5	5	32.8	48
E	37.6	4,900	2,100	27.7	1	27.7	48
C	33.3	5,200	1,800	26.2	4	26.4	34
H	25.4	5,250	1,750	21.1	^b 10	22+	-----
I	26.0	4,850	2,150	20.7	^b 5	21+	-----
K	25.4	3,550	3,450	18.2	^b 7	19+	-----

^a The constant C was taken as 2,200, the average of the values computed from the assumed general depth-porosity equation, $C = Dw \frac{P_0}{100 - P_0}$

^b Near faults.

In general, the finer-grained samples (those containing the smaller and flatter particles) have been compacted more than the coarser-grained samples. Inasmuch as the porosities show the relation to differences of rock texture or grain size that might have been expected on theoretical grounds, this test tends to further strengthen the proposed methods of correcting for dip and depth that were used.

The effects of differences of dip, depth, and rock texture upon porosity have been treated more or less quantitatively in the preceding paragraphs. However, the writer realizes fully that he has not established any quantitative relations by this treatment. Far more observations and experimental data than are now available would be required to determine the precise effect upon porosity of any one of the several variables involved. On the other hand, this method of treatment seems to show that the proposed explanation of a decrease of porosity with an increase of dip is not qualitatively improbable—that, within the range of porosities and dips examined, it leads to results that are of the correct order of magnitude.

If this proposed explanation is essentially correct, some interesting corollaries follow. For example, the porosity and thickness of a bed of shale decrease as the bed is tilted. This decrease of thickness represents a decrease of volume; it is not compensated by an increase in the length of the bed, for that would pull the rock particles apart and maintain the original porosity. That is, the steeply dipping beds have not been stretched by vertical uplift;⁸¹ they have been tilted and bent into a narrower compass without im-

portant change of length. In short, they have been subjected to horizontal compression. Another corollary is that, inasmuch as the thickness measured at right angles to the bedding depends upon the angle of dip, the thickness of the bed before deformation (the thickness needed for most stratigraphic studies) is the distance across the tilted bed measured in a vertical direction.⁸² (See fig. 2.) This corollary seems to be verified by field measurements of the varying thickness of shale formations in the area.⁸³ A third consequence of this interpretation is that large quantities of water were squeezed out of the shale as it was compressed, and this water must have escaped upward through fractures, joints, or pores, or laterally through interlaminated sandstone beds. Mead⁸⁴ has suggested that the deformation and dilatation of sediments [sandstones] may be an important factor in the movement of oil, gas, and water toward anticlines and monoclines. However, in view of the large proportion of shale interlaminated with the sandstone beds in most oil fields and the steep dips common in oil fields in the Rocky Mountain States, it seems more probable that in this region fluid movements were commonly away from rather than toward the areas of deformation.

SHALY STRUCTURE OR FISSILITY

One object of the microscopic study of the samples was to obtain some information on the nature of the

⁸² For field methods of measuring the vertical thickness see Ickes, E. L., The determination of formation thicknesses by the method of graphical integration. Am. Assoc. Petroleum Geologists Bull., vol. 9, pp. 451-463, 1925; Rubey, W. W., op. cit., pp. 339-348.

⁸³ Rubey, W. W., op. cit., pp. 333-351; Cretaceous and Cenozoic formations on the northwest flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [in preparation].

⁸⁴ Mead, W. J., op. cit., pp. 691, 697-698.

⁸¹ Rubey, W. W., Determination and use of thicknesses of incompetent beds in oil-field mapping and general structural studies: Econ. Geology, vol. 21, pp. 338-339, fig. 2, 1926.

thin laminations or planes of fissility which constitute the essential characteristic of shale. Many geologists seem to consider these laminations simply bedding planes.⁸⁵ Others, however, think that shaly structure is a secondary fissility only approximately parallel to the bedding. Thus Dana,⁸⁶ Grabau,⁸⁷ Cole,⁸⁸ Lewis,⁸⁹ and others have explained it as the result of the rotation of original flat flakes or the distortion or fracturing of irregular particles or the growth of micaceous crystals until the grains are essentially parallel to the bedding.

Field evidence indicates that secondary processes are at least a factor in the development of shaly structure in the Upper Cretaceous rocks in the Black Hills region. The planes of parting exposed in shale pits dug for contact dip readings are generally assumed to be identical with the planes of bedding and of fissility in the weathered shale. But, as many geologists who have done detailed structural mapping in Wyoming and Montana have no doubt noticed, the observed dips and strikes in two near-by pits or on two planes in one pit are rarely identical, and the planes of parting in some shale pits are inclined 5° or even more to thin layers of clay or sandstone which may be exposed. Observations of this sort were made, for example, on the flanks of the Pump Creek and Rocky Point anticlines, in T. 48 N., R. 64 W., and T. 56 N., R. 69 W., Wyoming. Field observations such as these indicate either that fissility is not everywhere strictly parallel to bedding or that a system of joints, subparallel to bedding and not uncommon in shale, may easily be confused with true fissility or bedding. Whichever of these two explanations is correct, it seems probable that some secondary process develops planes of parting roughly parallel to bedding in shale. By inference it also seems probable that at least some of the fissility of shale is caused by this secondary process.

Other field evidence also suggests a partly secondary origin of the fissility in the Upper Cretaceous shales of this region. The generally greater abundance of shaly structure in older rocks⁹⁰ holds in this region and suggests that fissility may be produced by the weight of overburden or the lapse of time. The Skull Creek and Belle Fourche members of the Graneros shale, near the bottom of the Upper Cretaceous section, contain much the greater part of the most

highly fissile shale in the Black Hills region. However, this evidence is not conclusive, for the Mitten member, near the middle of the Pierre shale, much higher in the stratigraphic section, also shows pronounced fissility.

The occurrence of some fissile shale high in the stratigraphic section suggests that shaly structure may be caused by differences in lithology. In fact, in the samples studied, for which chemical analyses are available, the fissility seems to vary inversely with the calcium carbonate content—precisely the opposite of the relation noted by Grabau.⁹¹ On the other hand, the clay content, or grain size, seems to have little if any relation to fissility. Of the samples that were mechanically analyzed the most fissile ones show the widest range of grain size: the Belle Fourche shale contains the most clay, the fissile middle member of the Pierre about the least, and the Skull Creek an intermediate amount. That is, although the calcium carbonate content may be a factor, the effect of other lithologic differences upon fissility is not clearly shown.

Microscopic examination of the shale samples likewise yielded only negative evidence on the origin of the shaly structure. Nearly all the cracks or joints in the thin sections studied follow the bedding as marked by layers of differing grain size or composition. However, a few cracks are inclined at low angles to the bedding, and if these cracks truly represent planes of fissility, they afford evidence that at least some of the fissility is secondary.

The planes of fissility or parting presumably follow the cleavage or orientation of the mineral particles in shale. These individual mineral particles are very small, and it is difficult or impossible to determine satisfactorily the direction of elongation of each grain. However, most of the crystals of clay minerals in the thin sections of shale examined lie so nearly parallel to bedding that they give the rocks an aggregate optical orientation (pp. 5-6). The supposition that the aggregate mineral orientation represents incipient fissility is strengthened by the fact that, in general, the most fissile specimens and those collected where the rocks dip most steeply⁹² show the most pronounced aggregate orientation. On the other hand, the aggregate orientation seems also to be related to the lithologic character of the rock, for it is more pronounced in the finer-grained and less calcareous specimens. The only conclusion that can be drawn from these observations with the microscope is that the shaly structure, if it follows the aggregate orientation, is essentially (but not necessarily exactly) parallel to the bedding.

⁸⁵ Geikie, Archibald, Textbook of geology, vol. 1, pp. 169-170, 1903. Chamberlin, T. C., and Salisbury, R. D., Geology, vol. 1, p. 487, 1909. Pirsson, L. V., Physical geology, p. 267, 1915; Rocks and rock minerals, p. 327, 1915. Harker, A., Petrology for students, p. 216, 1919. Hatch, F. H., and Rastall, R. H., The petrology of the sedimentary rocks, p. 200, 1923. Willis, Bailey, Geologic structures, p. 2, 1923.

⁸⁶ Dana, J. D., Manual of geology, p. 92, 1895.

⁸⁷ Grabau, A. W., Principles of stratigraphy, pp. 785-786, 1913.

⁸⁸ Cole, G. A. J., Rocks and their origins, pp. 83-84, 1922.

⁸⁹ Lewis, J. V., Fissility of shale and its relations to petroleum; Geol. Soc. America Bull., vol. 35, pp. 570-589, 1924.

⁹⁰ Lewis, J. V., op. cit., p. 581.

⁹¹ Grabau, A. W., Principles of stratigraphy, p. 785, 1913.

⁹² For evidence that the rocks have been deformed internally by tilting during earth movements see pp. 35-38, 51.

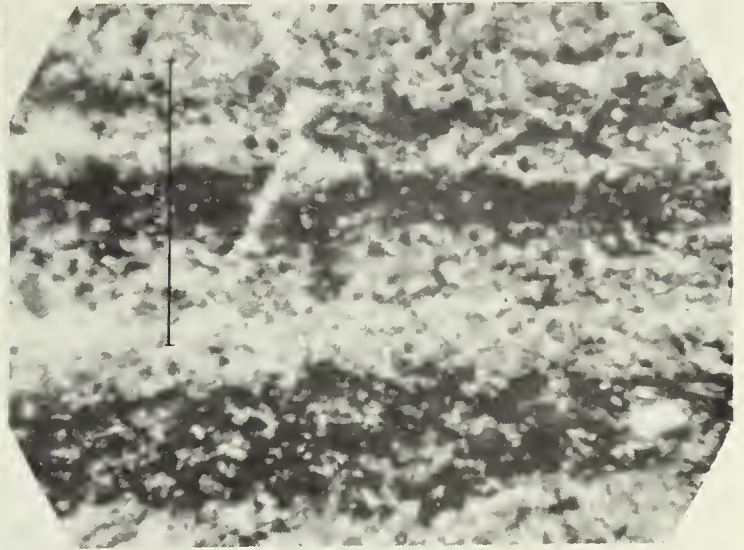
PLATE 4

1. Siliceous shale from Mowry shale member of Graneros shale in center of sec. 7, T. 48 N., 65 W., Weston County, Wyo. Laminations made by alternations of very fine light-gray silt and organic clay. Magnification shown by line 0.1 millimeter long.
2. Mudstone from lower part of Pierre shale in S. $\frac{1}{2}$ sec. 32, T. 49 N., R. 66 W., Crook County, Wyo. Laminations made by alternations of (*a*) very fine quartz sand and coarse silt and (*b*) dark clay. Magnification shown by line 1 millimeter long.
3. Black shale from lower part of Belle Fourche shale member of Graneros shale in north center of sec. 11, T. 48 N., R. 66 W., Weston County, Wyo. Laminations made by alternations of quartz silt and dark organic clay. Magnification shown by line 0.1 millimeter long.
4. Gritty shale from Carlile shale in W. $\frac{1}{2}$ sec. 35, T. 9 S., R. 61 E., Carter County, Mont. Laminations made by alternations of (*a*) very fine quartz sand and coarse silt and (*b*) dark clay. Magnification shown by line 1 millimeter long.
5. Black shale from upper part of Belle Fourche shale member of Graneros shale in W. $\frac{1}{2}$ sec. 34, T. 58 N., R. 62 W., Crook County, Wyo. Laminations made by alternations of light and very dark silt and clay. Magnification shown by line 0.1 millimeter long.
6. Calcareous marl from Greenhorn formation in SE. $\frac{1}{4}$ sec. 31, T. 45 N., R. 61 W., Weston County, Wyo. Laminations made by alternations of crystalline aggregates of calcium carbonate and dark clay. Magnification shown by line 1 millimeter long.

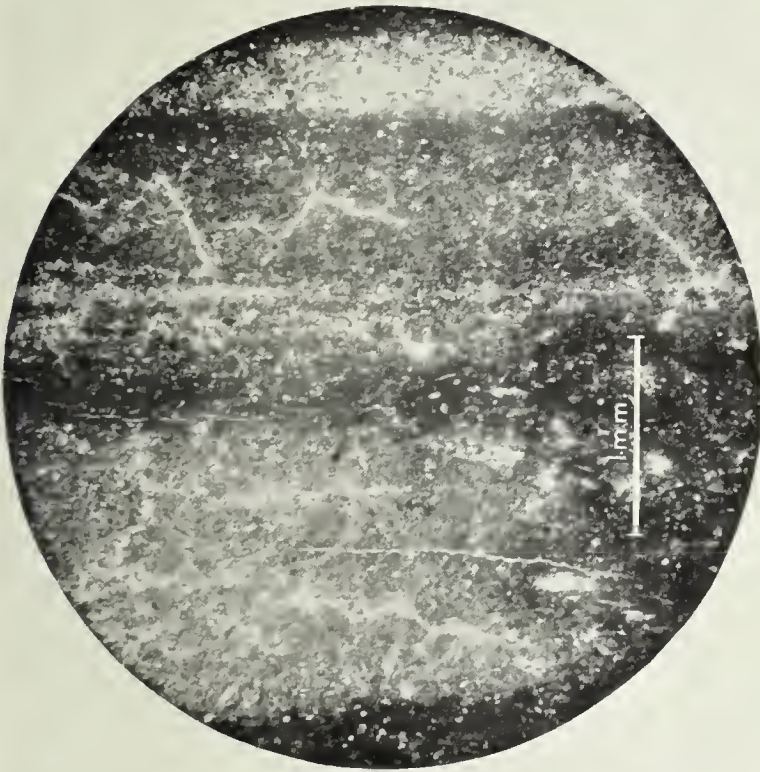
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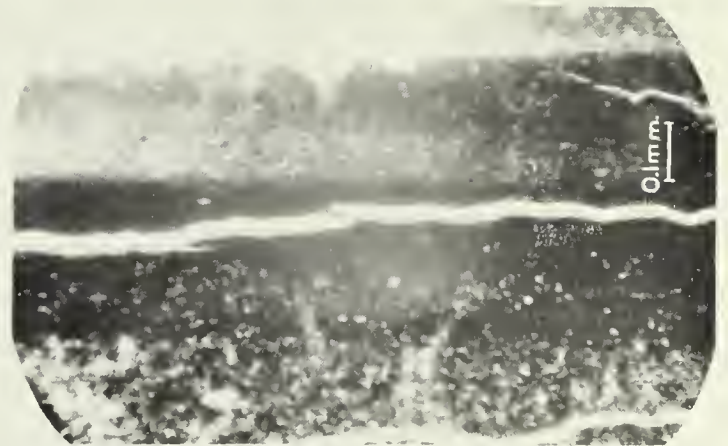
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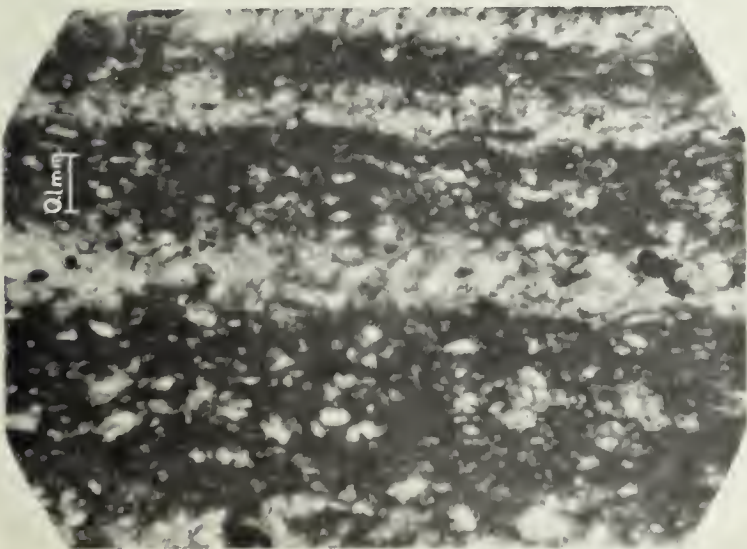
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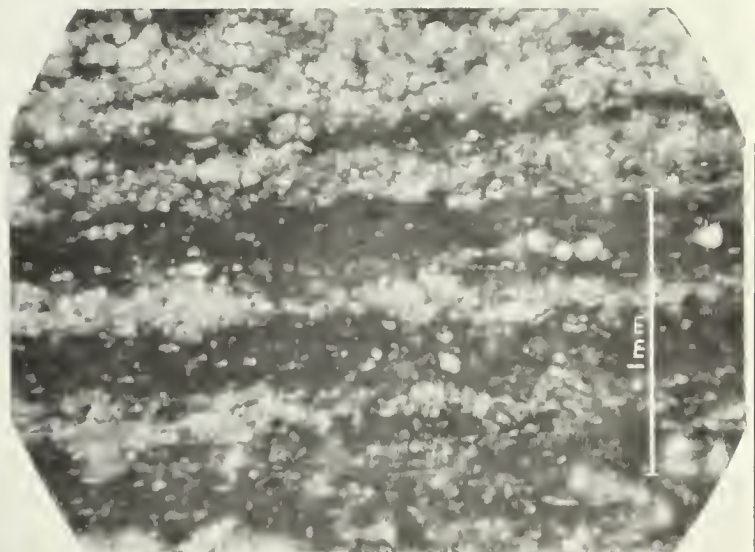
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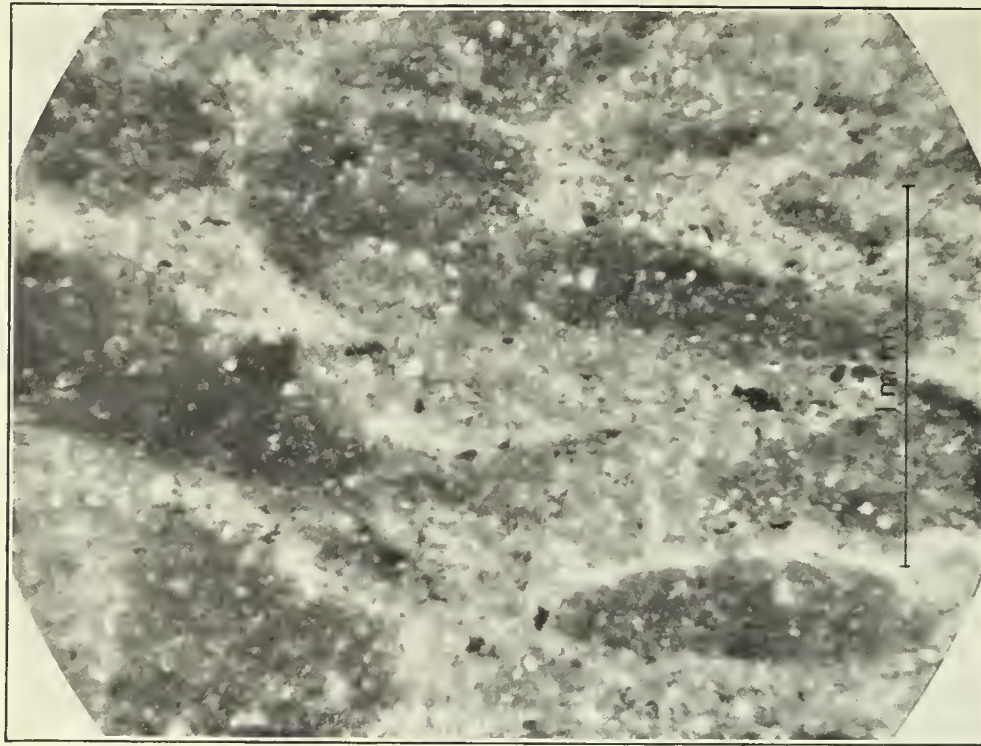
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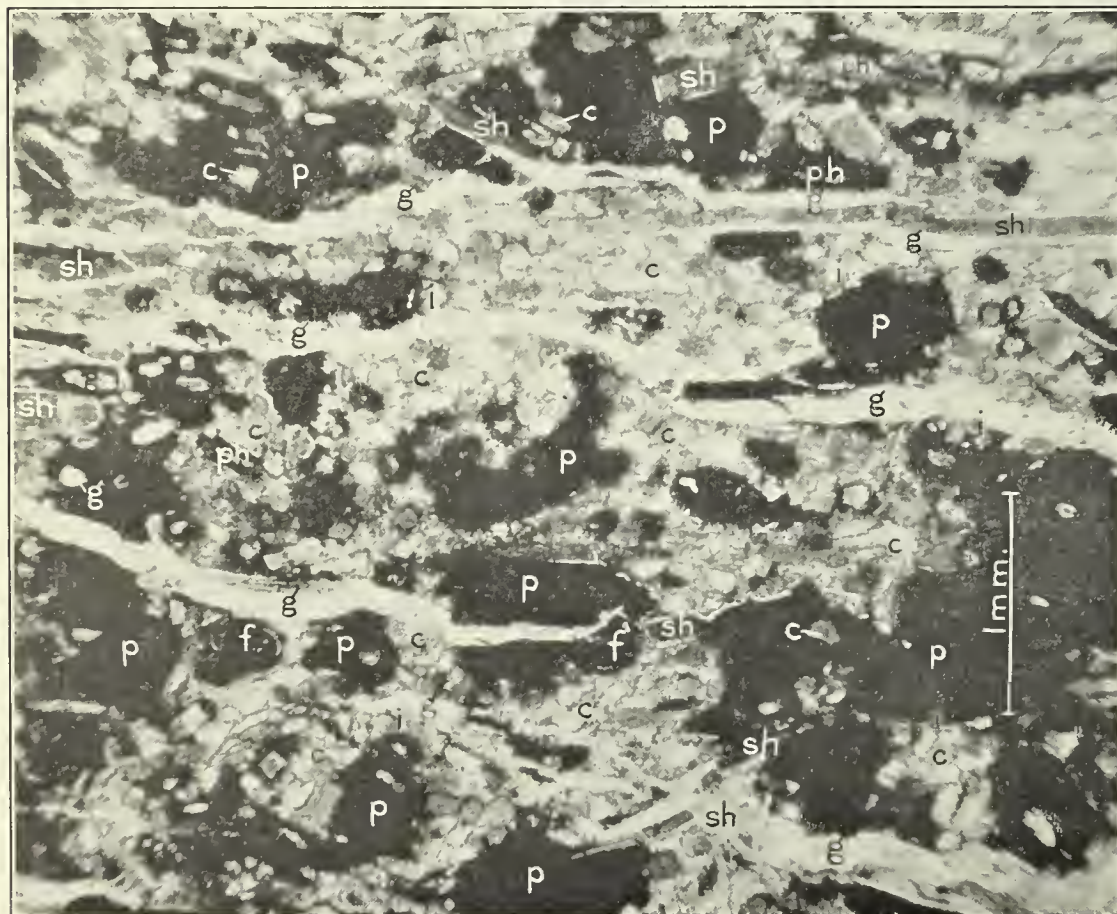


THIN SECTIONS SHOWING LAMINATIONS IN UPPER CRETACEOUS ROCKS OF THE BLACK HILLS REGION



A. MUDSTONE FROM UPPER PART OF PIERRE SHALE IN SEC. 21, T. 3 S., R. 62 E., CARTER COUNTY, MONT.

Brecciation while mud was still soft shown by minute crumpling of layers of dark clay in matrix of very fine quartz sand and coarse silt. Magnification shown by line 1 millimeter long.



B. PYRITIC LIMESTONE FROM THE BASAL BED OF THE GREENHORN FORMATION IN THE SW. ¼ SEC. 11, T. 57 N., R. 62 W., CROOK COUNTY, WYO.

Crystalline calcite (c), shells of *Inoceramus* (sh), and masses of minutely crystalline pyrite (p), cut by veinlets of microfibrous gypsum (g); shells (now gypsum) of Foraminifera (f), in the pyrite and iron oxide (i) bordering some of the pyrite masses. A few phosphatic fragments (ph) of bones and teeth. Magnification shown by line 1 millimeter long.

wave action commonly extends at 300 feet.⁴ These depths depend upon the grain size of the bottom sediment, the strength of winds, the distance from the shore (the "fetch" of the wind), and other factors.⁵ The Upper Cretaceous shales of the Black Hills region were deposited as fine-grained sediments in a sea that was open to the winds for many hundreds of miles; hence the conditions were favorable for a rather deep wave base. Yet ancient seas that invaded the continental platforms, such as the Upper Cretaceous interior area, are thought to have been generally rather shallow—only a few hundreds or tens of feet deep.⁶ Consequently, any annual laminations that might have formed could have been preserved only if the Upper Cretaceous sea was unusually deep throughout or locally near its center in the Black Hills region or if the winds that cause waves were not as strong in Upper Cretaceous time as now.

Some evidence indicates that the Upper Cretaceous sea was relatively deep in the Black Hills region. Shallow-water deposits are very uncommon in the Upper Cretaceous rocks of that region, and whatever the origin of the thin laminations, the mere fact that they were preserved suggests that the shales were deposited below effective wave base. The Upper Cretaceous rocks become much thicker westward from the Black Hills, but this greater thickness does not mean deeper water, for as they thicken shallow-water and continental deposits become more abundant.⁷ According to paleogeographic maps, the center of the Upper Cretaceous sea lay near the present Black Hills,⁸ and, in view of the structural and geographic relations at that time, it seems a reasonable assumption that the water was commonly deepest near the center of the sea.

Furthermore, wave action may not have extended as deep during Upper Cretaceous time as it does to-day. The depth of wave action depends upon the intensity of winds, and as winds are caused by differences in temperature and pressure of the atmosphere, their intensity is closely related to the diversity of climate.⁹ The fact that seas were widespread during Upper Cretaceous time in itself indicates that the climate then was rather moderate and geographically mi-

form,¹⁰ and the geographic distribution of fossil plants, vertebrates, and invertebrates also indicates relatively widespread equable climates in Upper Cretaceous time.¹¹

In summary: Annual laminae might conceivably have formed, for the Upper Cretaceous climate in the Black Hills region was probably sufficiently seasonal to cause annual laminations; and flocculation in salt water, although perhaps hindering the separation of coarse and fine layers, would not have prevented the formation of organic and lime-rich layers. Once formed, thin layers had some chances of preservation, for the sea was probably relatively deep near the present Black Hills, and wave base may have been relatively shallow at that time.

PROBABLE THICKNESS OF ANNUAL LAYERS

The hypothesis that the laminations are annual may be tested by evidence of another sort. Are the pairs of laminations too thick or too thin to be yearly deposits? The expected thickness of annual layers can not be calculated precisely, but the probable order of thickness can be estimated by several methods, and this can be compared with the observed thicknesses.

Many Pleistocene glacial-lake deposits in Europe and North America show varves (annual layers) marked by alternations of clay and silt in layers 10 millimeters or more thick.¹² Some recent and ancient lake deposits have annual laminations, and in places these laminations, which range from less than 0.1 to more than 10 millimeters in thickness, are marked by layers of organic matter and of calcium carbonate.¹³ Other kinds of sediments,¹⁴ including a few marine

¹⁰ Idem, p. 275.

¹¹ Schuchert, Charles, *Climates of geologic time*: Carnegie Inst. Washington Pub. 192, pp. 282-283, 1914. Knowlton, F. H., *A fossil flora from the Frontier formation of southwestern Wyoming*: U. S. Geol. Survey Prof. Paper 108, pp. 79-80, 1917; *Evolution of geologic climates*: Geol. Soc. America Bull., vol. 30, pp. 526-527, 1919. Stanton, T. W., *Evidence of invertebrates on the question of climatic zones during Mesozoic time*: Sci. Monthly, vol. 20, pp. 462-463, 1925.

¹² Antevs, Ernst, *The recession of the last ice sheet in New England*: Am. Geog. Soc. Research Ser. 11, 1922; *Retreat of the last ice sheet in eastern Canada*: Canada Geol. Survey Mem. 146, 1925; *Varved sediments*: Nat. Research Council Comm. on Sedimentation Rept., 1925-26, pp. 80-84, 1926; idem, 1926-27, pp. 53-59, 1927. Sauramo, Matti, *Studies on the Quaternary varve sediments in southern Finland*: Comm. géol. Finlande Bull. 60, 1923. Sayles, R. W., *Progress of studies on seasonal deposition of sediments*: Nat. Research Council Comm. on Sedimentation Rept., 1924, pp. 38-43, 1925.

¹³ Whittaker, E. J., *Bottom deposits of McKay Lake, Ottawa*: Roy. Soc. Canada Trans., 3d ser., sec. 4, pp. 141-156, 1922. Antevs, Ernst, *Retreat of the last ice sheet in eastern Canada*: Canada Geol. Survey Mem. 146, pp. 5-6, 1925; *Varved sediments*: Nat. Research Council Comm. on Sedimentation Rept., 1925-26, p. 82, 1926; idem, 1926-27, pp. 55-56, 1927. Bradley, W. H., *The varves and climate of the Green River epoch*: U. S. Geol. Survey Prof. Paper 158, pp. 95-97, 1929.

¹⁴ Antevs, Ernst, *Canada Geol. Survey Mem. 146*, pp. 5-7, 1925; Nat. Research Council Comm. on Sedimentation Rept., 1925-26, pp. 82-83, 1926.

⁴ Barrell, Joseph, *Factors in movements of the strand line and their results in the Pleistocene and post-Pleistocene*: Am. Jour. Sci. 4th ser., vol. 40, pp. 6-8, 1915; *Rhythms and the measurements of geologic time*: Geol. Soc. America Bull., vol. 28, pp. 779-780, 1917.

⁵ Barrell, Joseph, *Geol. Soc. America Bull.*, vol. 28, p. 779, 1917.

⁶ Schuchert, Charles, *Paleogeography of North America*: Geol. Soc. America Bull., vol. 20, p. 438, 1910. Barrell, Joseph, op. cit., pp. 768-769.

⁷ Reeside, J. B., jr., personal communication.

⁸ Schuchert, Charles, *Sites and nature of North American geosynclines*: Geol. Soc. America Bull., vol. 34, pp. 228-229, 1923.

⁹ Brooks, C. E. P., *Climate through the ages*, pp. 47, 63-64, New York, 1926.

deposits,¹⁵ have also been reported as annually laminated. The laminations in the marine shales range from 1 to 30 millimeters in thickness. (See table on p. 48.)

These are the thicknesses of reported varves or annual laminations in rocks from other regions, but it is possible by three independent methods to estimate very roughly the expected thickness of annual laminations in Upper Cretaceous rocks near the Black Hills. First, the average thickness of annual layers may be obtained by dividing the thickness of beds or alternations of beds deposited during some climatic or other cycle by the number of years in the cycle. Second, the average thickness of sediments laid down annually in a basin the size of the Upper Cretaceous interior sea may be calculated roughly from the rate of erosion in present-day drainage systems. Third, this average annual increment may be estimated by dividing the total thickness of Upper Cretaceous rocks by the number of years supposed to have elapsed during Upper Cretaceous time.

ESTIMATE BASED ON SUPPOSED RECORD OF PRECESSION CYCLE

In the Greenhorn limestone and Niobrara chalk of eastern Colorado G. K. Gilbert¹⁶ found a number of alternations of calcareous and argillaceous strata. He noted that the thickness of strata involved in these alternations increased with the proportion of shale in the rocks. As it seemed to him probable that the shale was deposited more rapidly than the lime, this suggested that the different alternations represent the deposits of some one uniform time cycle. The precession of the equinoxes (with an average net period of about 21,000 years¹⁷) seemed to Gilbert to be the cycle that would best account for these alternations. He took 4 feet as the thickness of the alternations in the noncalcareous shale, which in eastern Colorado makes up most of the Upper Cretaceous rocks. If his assumptions are correct, the annual layers in this shale would average about 0.06 millimeter thick. This estimate may be applicable in a rough way to the Black Hills region, for in both regions the Upper Cretaceous rocks are very similar in lithologic character and total thickness.

¹⁵ Heim, Albert, Einige Gedanken über Schichtung: *Naturforsch. Gesell. Zürich Vierteljahrshchr.*, vol. 54, pp. 331-332, 1909. Winkler, Artur, Untersuchungen zur Geologie und Paläontologie des steirischen Tertiärs: *K.-k. geol. Reichsanstalt Jahrb.*, vol. 63, p. 577, 1913. Sayles, R. W., The dilemma of the paleoclimatologists: *Am. Jour. Sci.*, 5th ser., vol. 3, pp. 471-472, 1922. Sauramo, Matti, op. cit., pp. 75, 109-129. Stamp, L. D., Seasonal rhythm in the Tertiary sediments of Burma: *Geol. Mag.*, vol. 62, pp. 515-528, 1925. Leighton, M. M., Studies of glacial sediments in 1926: *Nat. Research Council Comm. on Sedimentation Rept.*, 1926-27, p. 44, 1927. Marr, J. E., A possible chronometric scale for the graptolite-bearing strata: *Paleobiologica*, Jahrgang 1, Band 1, Teil 1, pp. 161-162, 1928.

¹⁶ Gilbert, G. K., Sedimentary measurement of Cretaceous time: *Jour. Geology*, vol. 3, pp. 121-127, 1895.

¹⁷ Idem, p. 124.

ESTIMATE FROM UPPER CRETACEOUS GEOGRAPHY AND PRESENT RATE OF EROSION

If the area of land draining into a settling basin of known size and the annual rate of erosion on this land are known, the average thickness of annual deposits in the settling basin may be calculated.

The probable location and extent of land and sea in North America during Upper Cretaceous time have been graphically shown by several geologists on paleogeographic maps. These graphic interpretations of Upper Cretaceous geography are based upon the present distribution, lithologic character, and fossil content of Upper Cretaceous sedimentary rocks and the known structural history of preceding periods. Paleogeographic maps are necessarily far from accurate, but they sum up existing knowledge into a most useful regional picture. According to these maps, during Upper Cretaceous time the seas encroached upon the present North American continent along the Pacific and Atlantic coasts, and a broad arm of the sea extended northward through the interior in the present region of the Rocky Mountains and Great Plains. This interior sea (the one in which the Upper Cretaceous rocks of the Black Hills region were deposited) joined the Atlantic Ocean through an enlarged Gulf of Mexico and was separated from the Pacific Ocean by a long, narrow range of highlands.

Paleogeographic maps of the Upper Cretaceous distribution of land and sea compiled by different geologists are very similar to one another, and from them the area of the lands that then underwent erosion and the area of the interior basin in which the eroded material was deposited can be estimated. For convenience in computation only those portions of the western highlands, the interior sea, and the eastern land mass that lay within the present limits of the United States may be considered, as the ratio of land to sea in this area probably indicates approximately the ratio of areas of erosion and deposition that influenced the thickness of sediments deposited annually in the Black Hills region. On four representative paleogeographic maps¹⁸ the area of the western highlands within the present United States is shown as approximately half a million square miles, and the areas of the interior settling basin (exclusive of the Mississippi embayment) and the eastern land mass as each slightly less than a million square miles.

How much of the areas of the western highlands and the eastern land mass drained into the interior sea is unknown, because parts of these lands sloped

¹⁸ Willis, Bailey, and Salisbury, R. D., *Outlines of geologic history, with especial reference to North America*, p. 198, Chicago Univ. Press, 1910. Pirsson, L. V., and Schuchert, Charles, *A textbook of geology*, p. 893, 1915. Grabau, A. W., *A textbook of geology*, p. 704, 1921. Schuchert, Charles, Sites and nature of North American geosynclines: *Geol. Soc. America Bull.*, vol. 34, pp. 228-229, 1923.

toward the Pacific, Atlantic, and Gulf of Mexico. The simplest assumption is that on each of these land areas the drainage divide lay midway between the bordering seas and that one-half of each land area drained into the interior sea. This assumption probably gives an unduly small drainage area tributary to the interior sea, because on the eastern land mass the divide probably lay near the Appalachian Mountains, far east of the center; and furthermore a part of the large land mass in eastern Canada probably drained southwestward into the United States. Although the probability of this error in the estimate can be recognized, it is difficult to establish quantitatively the needed correction. For that matter, as the estimate is necessarily an approximate one, such a correction is hardly worth attempting.

Another uncertainty is the average area of the interior sea during Upper Cretaceous time. It is true that the paleogeographic maps may not show the maximum extent of the seas during a period or even at any one time. On the other hand, the shore lines undoubtedly shifted constantly, and the average size of the interior sea during Upper Cretaceous time was probably much less (and the average size of the land areas correspondingly greater) than the size of the sea at the time of its maximum extent. At the beginning and end of the Upper Cretaceous invasion the interior basin was above sea level, and at times during the Upper Cretaceous epoch parts of the basin underwent erosion. However, despite the uncertainty from this constant shifting, it may be safe to assume that, on the average, the interior sea was more than one-half the size shown on the maps.

Hence, taking the areas of land and sea within the limits of the United States as shown on paleogeographic maps and assuming that one-half the drainage from these lands was tributary to the interior sea, we may estimate that the average ratio of erosion areas to deposition areas throughout Upper Cretaceous time was probably between $\frac{800,000}{1,000,000} = 0.8$ and $\frac{800,000 + 500,000}{1,000,000 - 500,000} = 2.6$. If, as seems likely, somewhat more than half of the adjacent land areas drained into the interior sea, both of these ratios should be increased. Although subject to considerable error because of these uncertainties, an estimate that the areas furnishing sediment averaged slightly larger than the area of deposition (perhaps $\frac{1,000,000}{750,000} = 1.3 \pm$) may be taken as a rough approximation to the average ratio.

Materials eroded from the land are carried away in chemical solution in stream water and by mechanical suspension and rolling along the stream bottom. The portion carried in solution may eventually be

distributed throughout the ocean, but the sediment transported mechanically into marine water is nearly all deposited within a few hundred miles of the stream mouths. Of the mechanical load the material rolled along the bottom is coarse grained and deposited relatively near shore, but the suspended sediment is fine grained and makes widespread mud deposits. Consequently, the suspended sediment brought from adjacent lands each year would afford a minimum but perhaps the best available estimate of the average thickness of annual deposits over the entire interior Upper Cretaceous sea.

The volume of detritus eroded each year from a unit area of land varies from year to year and from place to place. The yearly amount and seasonal distribution of rainfall and run-off, the resistance of outcropping rocks to erosion, and the slope of the land are some of the chief factors affecting this rate of erosion. The average annual rate of erosion in many different drainage basins has been determined approximately by engineers and found to vary greatly from one basin to another. In order to estimate the erosion rate on the Upper Cretaceous land areas, drainage basins in which present conditions are roughly comparable to those of Upper Cretaceous time should be chosen; but this choice is exceedingly difficult because only the most general facts are known about the climate and topography of these Upper Cretaceous land areas.

The drainage basin of the Mississippi River is perhaps as good for comparison as any other, because, despite the fact that erosion is somewhat more rapid in this basin than in most other parts of the United States, it is very large and embraces a wide variety of the conditions that affect erosion, its rate of discharge of eroded material has been rather carefully determined, and it lies to-day in the same part of the world as the Upper Cretaceous land masses under consideration. The quantity of suspended sediment carried into the Gulf of Mexico annually by the Mississippi River has been estimated at 340,500,000 tons,¹⁹ and the coarse material rolled along the bottom of the river at about one-tenth as much.²⁰ The specific gravity of the particles making up the suspended load is approximately 2.6,²¹ and the drainage area of the river is about 1,265,000 square miles.²² Therefore material equivalent to a layer of solid rock averaging about 0.0014 inch or 0.036 millimeter in thickness is eroded off the surface of the land each year and carried in suspension into the Gulf of Mexico. If, instead of being solid rock, the eroded

¹⁹ Dole, R. B., and Stabler, Herman, Denudation: U. S. Geol. Survey Water-Supply Paper 234, p. 84, 1909.

²⁰ Humphreys, A. A., and Abbott, H. L., Report on physics and hydraulics of Mississippi River, p. 149, 1861.

²¹ Dole, R. B., and Stabler, Herman, *op. cit.*, p. 80.

²² *Idem*, p. 84.

material contains 50 per cent of pore space, the layer removed each year averages 0.072 millimeter in thickness.

These approximate figures—the relative areas of land and sea during Upper Cretaceous time and the thickness of mud-forming detritus eroded annually—afford a basis for estimating the average thickness of annual shale deposits in the Upper Cretaceous interior sea. In nine samples of Upper Cretaceous shale from the Black Hills region the porosity ranges from 24 to 38 per cent and averages 30 per cent. (See pp. 32, 34.) The probable drainage and deposition areas indicate that an annual layer of solid silt about 0.05 millimeter thick, or, if the material has a porosity of 30 per cent, a layer 0.07 millimeter thick would be expected.

This estimate is of course only approximate; it means that the thickness of annual layers is probably to be measured in tenths or hundredths of millimeters, not in millimeters or centimeters, nor, on the other hand, in microns or millimicrons. Even should 0.07 millimeter be the correct average thickness for the entire sea, the thickness of the layers would certainly vary from place to place. Mud deposits near shore might be thicker and those farthest from land thinner than the average. However, as applied to the Black Hills region, many of the possible errors in the estimate tend to compensate one another. The possibly excessive annual thickness estimated by using the Mississippi River rather than some other drainage system as a basis of computation is probably more than offset by using too small a drainage area tributary to the interior sea and by disregarding the coarser material brought in by streams. Similarly the great distance of the Black Hills region from the shores of the Cretaceous sea and the consequent thinness of deposits that might be expected there is perhaps offset by the ignored organic and chemical deposits, such as calcium carbonate and bases adsorbed on the surfaces of fine clay particles—deposits that might be thicker in the middle of the settling basin than near shore. Furthermore, this expected thinness due to the central position of the Black Hills would be largely offset by the reworking of shore deposits. That is, present outcrops show that sediments did not long accumulate in excessive thickness near the eastern shore of the Upper Cretaceous sea. Therefore, as the floor of the eastern half of this settling basin was probably gently shelving, any temporarily excessive deposits near shore must have been reworked by waves and distributed westward.

ESTIMATE FROM TOTAL THICKNESS OF ROCKS AND SUPPOSED DURATION OF UPPER CRETACEOUS TIME

If the total thickness of rocks that accumulated during Upper Cretaceous time and the number of

years required for this accumulation are known, the average thickness of annual layers may be estimated. The writer's measurements of outcropping rocks and his correlations of well logs indicate that the total thickness of Upper Cretaceous rocks (excluding the Eocene (?) Lance formation) on the western flank of the Black Hills is about 4,000 or 4,500 feet, of which all but a few hundred feet are marine shales, mudstones, and marls.²³ In southwestern Wyoming, 250 miles from the Black Hills, the maximum thickness of rocks of the same age is more than 20,000 feet,²⁴ but there a large proportion of the total is made up of rather coarse continental deposits, which very probably accumulated more rapidly.

The time required for the deposition of these rocks is much more difficult to determine than their thickness. Almost all estimates that have been made of the duration of Upper Cretaceous time have been calculated by allotting to the Upper Cretaceous epoch a proportionate part of the time since the earliest geologic record. The basis for the proportionate allotment is indicated below. Estimates of the number of years that have elapsed since the earth reached essentially its present condition have been made by widely different methods, and the estimates themselves have also varied widely. The principal criteria have been the time required for the development of life forms, for the loss of heat by the earth and sun, for the denudation of lands and accumulation of sediments, for the number of mountain-building revolutions recorded in the rocks, for the concentration of salt in the sea, and for the disintegration of radioactive minerals.

Of these criteria the two that are generally thought to afford most definite estimates are those based on salt in the ocean and on lead-uranium ratios. But these two, though most definite, give very different results. The quantity of sodium in the ocean divided by the quantity now being added each year (with some minor corrections) gives somewhat less than 100,000,000 years²⁵ as the age of the ocean. The ratio of the quantity of the unstable element uranium to the quantity of its disintegration product, lead, with which it is found associated, and the observed rate of radioactive disintegration of uranium indicate that the oldest rocks exposed on the earth's surface are more than 1,000,000,000 years old.²⁶ Thus, though it

²³ Rubey, W. W., Cretaceous and Cenozoic formations on the northwest flank of the Black Hills: U. S. Geol. Survey Prof. Paper — [in preparation]. (See also pp. 3-5 of this report.)

²⁴ Wilmarth, M. G., The geologic time classification of the United States Geological Survey compared with other classifications: U. S. Geol. Survey Bull. 769, p. 6, 1925; Tentative correlation of geologic formations in Wyoming (mimeographed chart), U. S. Geol. Survey, 1925.

²⁵ Clarke, F. W., The data of geochemistry, 5th ed.: U. S. Geol. Survey Bull. 770, pp. 150-155, 1924.

²⁶ Holmes, Arthur, and Lawson, R. W., Factors involved in the calculation of the ages of radioactive minerals: Am. Jour. Sci., 5th ser., vol. 13, p. 342, 1927.

seems that these two estimates should be approximately the same, one is more than ten times as great as the other.

The relative merits of these and other estimates have been much discussed, and efforts have been made to reconcile them. Barrell²⁷ pointed out that because of the exceptional height and area of continents to-day the rate at which sodium is now being added to the ocean may be many times greater than it has been throughout the geologic past. Chamberlin²⁸ called attention to the acceleration of erosion in drainage basins occupied by human beings, to the deposition of some oceanic sodium by adsorption or base exchange on clay particles, and to reasons for considering chlorine, the accumulation rate of which indicates a greater age than sodium, as the better fitted of the two elements to serve as a criterion of age. On the other hand, Joly²⁹ contends that several lines of evidence indicate that uranium disintegrated more rapidly in the past than it does to-day. Critical discussion of these and other estimates is outside the scope of this paper. For the purpose of this investigation the estimates based on the sodium and uranium methods may suffice to indicate low and high figures for the approximate duration of geologic time.

There is much closer agreement among different writers about the proportional distribution of geologic time among the different eras and periods. The proportions of total time represented by the several divisions have been estimated chiefly from the volumes and maximum thicknesses of sediments of the respective ages. The percentage of Paleozoic and later time assigned to the Upper Cretaceous in a group of estimates summarized by Barrell³⁰ ranges between 4½ and 9 per cent. A recent compilation³¹ indicates that the maximum thickness of the Upper Cretaceous is 6.6 per cent of the total of the maximum thicknesses of Paleozoic and later rocks.

Considering both the supposed total duration of geologic time and the proportional distribution among the several periods, different geologists have estimated the duration of the Upper Cretaceous epoch at two to two and three-fourths million years on the basis of the sodium time scale, or forty to fifty million years on the basis of the uranium time scale,³² as well as making other estimates between these figures.

The thickness of Upper Cretaceous rocks on the western flank of the Black Hills divided by two or two and three-fourths million gives about 0.5 or 0.6 milli-

meter as the thickness of annual layers. Dividing by forty or fifty million gives a thickness of 0.025 or 0.030 millimeter for the annual layers. The thicker Upper Cretaceous section in southwestern Wyoming would, by this method, indicate annual layers several times thicker, but there the rocks are coarser grained and presumably accumulated more rapidly than those of the Black Hills region.

This method involves an assumption that none of the Upper Cretaceous rocks are missing from the stratigraphic section in the Black Hills region, and it therefore gives a minimum estimate of the thickness of annual layers. In a general way this assumption may be justified; no large unconformities within the Upper Cretaceous series are known in this region, and the paleontologic evidence indicates that nearly every faunal zone is represented.³³ However, field evidence suggests that there were in Upper Cretaceous time at least two periods of nondeposition or erosion (probably without uplift above sea level),³⁴ and perhaps many smaller stratigraphic breaks that have not been found. Also the Telegraph Creek fauna³⁵ of Montana has not been recognized near the Black Hills, and rocks of that age may be absent there. Any such gaps in the Black Hills Upper Cretaceous section would make the estimated thickness of annual layers too small. Although for this reason the estimate is too low, no one can say whether it should be increased by one-tenth or multiplied by 10.

It is of interest to note in passing that the minimum thickness of 0.025 or 0.030 millimeter based upon radioactive disintegration is only one-half or one-third of that based upon present rates of erosion (0.07). That is, under the assumption made, erosion may have been only one-third or one-half as rapid (or, if stratigraphic breaks are important, even more) in Upper Cretaceous time as it is now—not one-tenth, one-fifteenth, or one-twentieth, as, because of the unusual height of the continents to-day, Barrell³⁶ thought probable for most of the geologic past.

COMPARISON OF ESTIMATES WITH OBSERVED THICKNESS OF LAMINATIONS

Three totally independent methods of estimation indicate that, if annual layers are present in the Upper Cretaceous shales of the Black Hills region, they are probably a few tenths or hundredths of a millimeter thick (see table on p. 48), and, as the observed pairs of laminations average about 0.2 millimeter thick, the hypothesis that they may be annual layers is thereby strengthened. It is true that the

²⁷ Barrell, Joseph, Rhythms and the measurements of geologic time: *Geol. Soc. America Bull.*, vol. 28, pp. 834-838, 871-872, 1917.

²⁸ Chamberlin, T. C., The age of the earth from the geological viewpoint: *Am. Philos. Soc. Proc.*, vol. 61, pp. 252, 266-270, 1922.

²⁹ Joly, John, The surface history of the earth, pp. 150-153, 1925.

³⁰ Barrell, Joseph, *op. cit.*, pp. 884-885.

³¹ Wilmarth, M. G., *op. cit.* (Bull. 769), p. 6.

³² Barrell, Joseph, *op. cit.*, pp. 884-885. Wilmarth, M. G., *op. cit.*, p. 5.

³³ Reeside, J. B., jr., personal communication.

³⁴ Rubey, W. W., *op. cit.*

³⁵ Reeside, J. B., jr., A new fauna from the Colorado group of southern Montana: *U. S. Geol. Survey Prof. Paper* 132, pp. 25-31, 1923.

³⁶ Barrell, Joseph, *op. cit.*, pp. 747, 761, 774-776, 891-893, 1917.

observed pairs of laminations are about three times as thick as annual layers would be expected to be from estimates based (1) on G. K. Gilbert's supposed record of precession cycles and (2) on Upper Cretaceous geography and present erosion rates. Also they are perhaps seven or eight times as thick as the minimum thickness of annual layers computed from estimates of Upper Cretaceous time based on radioactive disintegration but only about one-third as thick as the minimum thickness computed from estimates based on ocean salt. However, although most of the estimates seem to indicate that the observed pairs of laminations are slightly thicker than might be expected, yet it is doubtful if any of the estimates are more accurate than the differences between the observed and computed thicknesses.

Thickness of laminations in sedimentary rocks of Black Hills and other regions

	Thickness (millimeters)	
	Range	Average
Pairs of laminations observed in Upper Cretaceous shale of Black Hills region.....	0. 05- 1. 25	0. 2
Annual laminations in sediments from other regions:		
Glacial varves.....	10 - 100	^a 40
Lacustrine varves.....	0. 1 - 10	^a 1
Marine varves.....	1 - 30	^a 2
Annual layers in Upper Cretaceous shale of eastern Colorado, computed from G. K. Gilbert's supposed record of precession cycle.....		^a 0. 06
Annual layers in Upper Cretaceous shale of Black Hills region, estimated from—		
Upper Cretaceous geography and present rate of erosion.....		^a 0. 07
Total thickness of Black Hills Upper Cretaceous rocks divided by supposed duration of Upper Cretaceous time:		
Cretaceous time = 2 million years (based on salt in ocean).....		^b 0. 60
Cretaceous time = 50 million years (based on lead-uranium ratios).....		^b 0. 03

^a About.

^b More than.

The estimates certainly suggest that the thickness of the pairs of laminations is of the correct order of magnitude for annual layers. Tides and daily variations of temperature and wind, which might possibly produce recognizable alternations in the sediments, would probably make layers only hundredths or thousandths as thick. Climatic cycles with periods longer than one year also seem to be unsatisfactory explanations of the laminations. Short ones like the sunspot cycle³⁷ do not now and perhaps never did cause

³⁷ Barrell, Joseph, op. cit., p. 825. Huntington, Ellsworth, and Visher, S. S., Climatic changes, their nature and causes, Yale University Press, 1922. Brooks, C. E. P., Climate through the ages, pp. 96-113, 1926.

as great variations as the annual contrast between summer and winter, and so of the two the annual cycle seems to offer a much more likely explanation. The much longer cycle of the precession of the equinoxes may cause very pronounced climatic variations, but any alternations thus produced would be many thousands of times as thick as the observed laminations. Storms recurring several times a year or every few years are not eliminated by these estimates of the probable thickness of annual layers; and the type of lamination marked by alternations of grain size might be so explained. However, as discussed on pages 40-41, these laminations marked by alternations of grain size apparently were formed in much the same way as the equally common organic laminations, which were probably not storm-made; and the preservation of thin layers indicates that storms rarely disturbed the bottom muds.

RATE OF ADVANCE OF UPPER CRETACEOUS SEA

The discussion thus far has led to the conclusions that annual laminations might possibly have been formed and preserved in the Upper Cretaceous rocks of the Black Hills region, that the observed laminations in these rocks are of different kinds that can be explained most simply as annual layers, and that they are of about the right thickness to be annual layers. These conclusions, of course, do not constitute proof that they are annual layers. In fact, more detailed comparisons suggest that they may have formed every few years instead of every year, although the relatively small discrepancies found in these comparisons can be explained equally well in other ways.

If the observed laminations are annual and if their average thickness is typical of the series in the region, they furnish a rough measure of the number of years that elapsed while certain events occurred. Applied in this way, they indicate that the Upper Cretaceous sea advanced eastward from the Black Hills rather rapidly.

Eastward and southward from the Black Hills the sandstone beds at or near the base of the Upper Cretaceous, which have generally been called Dakota sandstone, seem to transgress upward in the stratigraphic section.³⁸ In Nebraska, Kansas,³⁹ eastern Colorado, and eastern Wyoming these sandstone beds, which are largely nonmarine, underlie the marine Graneros shale, which in turn underlies the Greenhorn limestone. The Greenhorn limestone was probably deposited almost simultaneously throughout this area, yet the underlying Graneros shale thins from about 1,250 feet on the northwestern flank of the

³⁸ Grabau, A. W., Principles of stratigraphy, p. 739, 1913.

³⁹ Rubey, W. W., and Bass, N. W., The geology of Russell County, Kans.: Kansas Geol. Survey Bull. 10, pp. 62-63, 1925.

Black Hills⁴⁰ to about 40 or 60 feet in Kansas⁴¹ and eastern Nebraska.⁴² That is, the sandstone beds that underlie the Graneros shale apparently rise about 1,200 feet stratigraphically in the 450 miles between northeastern Wyoming and the vicinity of Sioux City, Iowa.

If this difference in the thickness of the Graneros shale was caused by the eastward transgression of the sandstone shore phase, so that the shale 50 feet below the Greenhorn in Wyoming was laid down far off shore at the same time as the top of the Dakota in

graphic breaks, then the sea advanced eastward across South Dakota in about 2,000,000 years (the number of feet multiplied by 1,500, the average number of laminations to the foot), or at an average rate of slightly more than 1 foot a year. Of course, even under ideally uniform conditions the rate of advance would not be constant from year to year. For example, as the sea advanced its area would increase and the area of dry land would decrease, so that the quantity of detritus spread over a unit area of the sea would become less each year. However, it does not

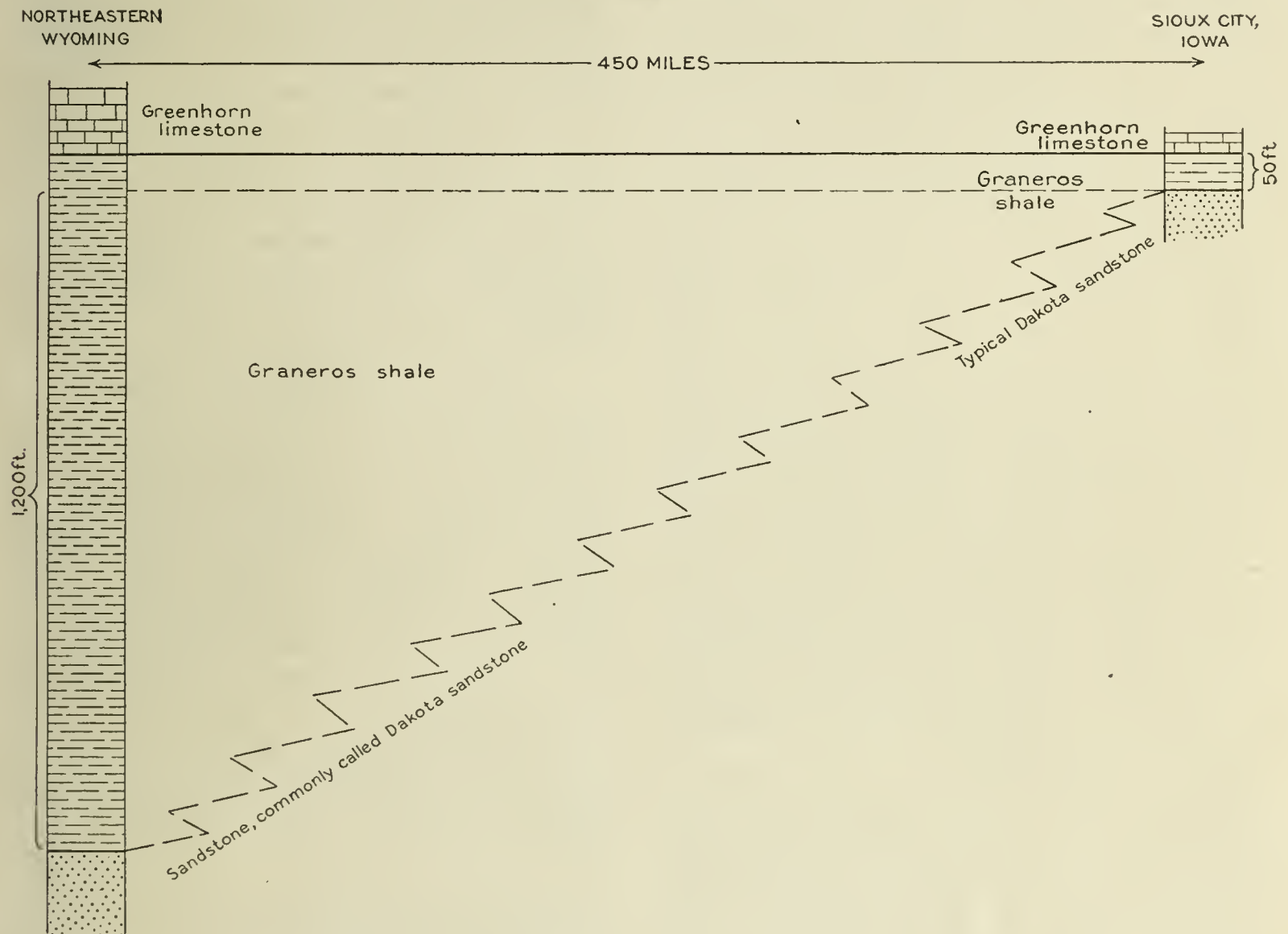


FIGURE 3.—Methods of estimating rate of eastward advance of Upper Cretaceous sea

Iowa, then the time required for the underlying 1,200 feet of Graneros to accumulate was the time required for the shore of the sea to advance the 450 miles. (See fig. 3.) If the laminations are annual and if the 1,200 feet of Graneros shale contains no strati-

necessarily follow that the laminations would become thinner and the rate of advance slower from year to year. If the sea floor did not sink as rapidly as detritus was delivered to it, the sea would nearly fill with sediment, and waves would sweep later detritus on out to deeper water.⁴³

For comparison with the foregoing estimate, the rate of advance may be computed on the same interpretation of the age relations of the Dakota sandstone but without using the laminations as a measure

⁴⁰ Rubey, W. W., Cretaceous and Cenozoic formations on the north-west flank of the Black Hills: U. S. Geol. Survey Prof. Paper— [In preparation].

⁴¹ Rubey, W. W., and Bass, N. W., op. cit., p. 51.

⁴² Condra, G. E., Geology and water resources of a portion of the Missouri River valley in northeastern Nebraska: U. S. Geol. Survey Water-Supply Paper 215, p. 11, 1908. Burchard, E. F., Geology of Dakota County, Nebr., with special reference to lignite deposits: Sioux City Acad. Sci. and Letters Proc., vol. 1, p. 150, 1904.

⁴³ Barrell, Joseph, op. cit., p. 748.

of time. If the estimates of geologic time based on the salt in the ocean are correct and the entire Upper Cretaceous epoch lasted only 2,000,000 years, then the 1,200 feet of Graneros shale, about one-third or one-fourth of the total thickness of Upper Cretaceous rocks (about 4,250 feet), accumulated in approximately 500,000 years, and the sea advanced about 4 or 5 feet a year. If the Upper Cretaceous epoch lasted about 50,000,000 years as indicated by lead-uranium ratios, then the sea probably advanced about 2 inches a year.

It is worth noting that the interpretation of the age relations of the Dakota sandstone on which these estimates are based permits the slowest possible advance of the sea. If, for example, the sea did not transgress eastward at a uniform rate but by a series of minor advances and retreats, then during the minor advances the shore line moved much more rapidly than the average rate. Or, again, if the difference in thickness of the Graneros shale was caused not by transgression of the sandstone eastward but by more rapid deposition of the shale toward the west—that is, if the sandstone was not transgressive but of the same age in Wyoming and Iowa—then the sea advanced so much more rapidly that the rate could not even be estimated. Or, third, the difference in thickness may have been caused partly by transgression eastward and partly by more rapid deposition westward, so that the Newcastle sandstone of Wyoming, several hundred feet above the base of the Graneros shale, may have been contemporaneous with the upper part of the Dakota sandstone of Iowa.⁴⁴ On this third interpretation the shore line moved across South Dakota during the deposition of about 200 instead of 1,200 feet of shale, and the average rate of advance must have been about six times as fast as the rates estimated from the first interpretation.

All these estimates of the rate at which the Upper Cretaceous sea spread over the land—even the one based on the lead-uranium ratios—seem rather high. The average rate of marine invasion on sinking coasts to-day is not known. In areas thought to be sinking very rapidly, such as the Netherlands, New Jersey, and northern Gulf of Mexico coasts,⁴⁵ the annual rate of vertical sinking has been estimated at as much as several millimeters, but the average rate for all sinking coasts must be much less. Some coasts are being cut back by waves several feet a year,⁴⁶ but Geikie⁴⁷ estimated that, on the average, this rate is much less than one-tenth of a foot a year. It is true that the

rate of coast retreat by submergence, which depends upon the rate of sinking and the slope of the land, is not the same as the rate of cutting by waves, yet from the rather common absence of exceedingly rough topography below unconformities, it seems probable that submergence has not often greatly exceeded wave erosion and that the present rate of cutting may be taken as a rough measure of the rate of submergence in the past. The land surface over which the Upper Cretaceous sea advanced was probably rather smooth and gently sloping,⁴⁸ hence its submergence may have been somewhat more rapid than that of the more rugged, steeply sloping land surfaces of to-day. That is, the laminations suggest that the Upper Cretaceous sea may have advanced at least ten times as rapidly as the present average rate of shore advance. This may mean that the laminations are not annual but that they formed every 10 years or so. However, considering the rapid advance indicated even without using the laminations as a measure of time, it seems more likely to mean that the Upper Cretaceous sea advanced more rapidly than most present-day seas, perhaps because it came in over a relatively flat land surface.

LENGTH OF UPPER CRETACEOUS TIME

The observed thickness of pairs of laminations has been compared with the reported thicknesses of annual laminations from other regions and with the expected thicknesses, as estimated by several different methods, of annual layers in the Black Hills region. This general comparison indicated that the paired laminations were of about the right thickness to be annual layers. Hence a more detailed comparison with one of the several methods of estimation used before is justified.

If the observed layers are typical of each formation in the Black Hills region, the total duration of Upper Cretaceous time indicated by the formation thicknesses is about five or ten million years. This estimate is greater than the two or two and three-fourths million years based on the salt in the ocean and less than the forty or fifty million years based on the lead-uranium ratios. The five or ten million years is, however, a minimum estimate, for it ignores the field and paleontologic evidence of stratigraphic breaks within the Upper Cretaceous series of this region. If 1,000 feet of Upper Cretaceous rock was eroded during Upper Cretaceous time, the estimates should be increased by about one-fourth.

If, as many geologists and geophysicists believe, the lead-uranium ratios are the most accurate measure of geologic time now available, and if forty or fifty million years is therefore the most probable duration of

⁴⁴ Stanton, T. W., Some problems connected with the Dakota sandstone: *Geol. Soc. America Bull.*, vol. 33, pp. 264-269, pl. 4, 1922.

⁴⁵ McGee, W. J., The Gulf of Mexico as a measure of isostasy: *Am. Jour. Sci.*, 3d ser., vol. 44, pp. 179-180, 187-188, 1892.

⁴⁶ Gregory, H. E., The formation and distribution of fluvial and marine gravels: *Am. Jour. Sci.*, 4th ser., vol. 39, p. 489, 1915. Johnson, W. D., Shore processes and shore-line development, pp. 69-71, 1919.

⁴⁷ Geikie, Archibald, *Textbook of geology*, vol. 1, p. 593, 1903.

⁴⁸ Lee, W. T., Reasons for regarding the Morrison an introductory Cretaceous formation: *Geol. Soc. America Bull.*, vol. 26, pp. 311-312, 1915. Rubey, W. W., and Bass, N. W., *op. cit.*, pp. 57-62, pl. 3.

Upper Cretaceous time, then the laminations indicate that only about one-fifth or one-tenth of total Upper Cretaceous time is represented in the rocks now present in the Black Hills region. As the Upper Cretaceous rocks in this region make a relatively complete stratigraphic section, this discrepancy leads almost certainly to one of four conclusions: (1) the laminations represent cycles longer than the present year; or (2) the average thickness of the observed laminations is not typical of the series in this region; or (3) the Upper Cretaceous epoch was much shorter than forty or fifty million years; or (4) inconspicuous stratigraphic breaks, aggregating four-fifths or nine-tenths of all Upper Cretaceous time, are distributed about evenly through the Upper Cretaceous section of the region.

It is possible that the duration of the year—the period of the earth's revolution about the sun—has varied during geologic time. It is also possible that climatic cycles of several years' length, such as the sun-spot cycle, may account for the discrepancy between the number of laminations and the estimated duration of the Upper Cretaceous epoch in years; but the contrast between summer and winter seems a more probable cause of alternations in marine sediments than the relatively slight variations during longer cycles. Again it is possible that an occasional storm destroyed the laminations of many years' accumulation, and that this disturbed sediment was redeposited after the storm as a single layer; that is, because of occasional storms, fewer layers might be preserved than the number of years required for the formation of the deposit. However, as discussed on pages 40–41, this explanation would not account for the layers of much and little organic matter, and it seems probable that the layers of different types were formed by the same process and during approximately equal intervals of time; also the thinness of the laminations and the relative uniformity of this thinness suggest that the bottom muds were never disturbed by storm waves.

The average thickness of the observed laminations may not be typical of the laminations throughout the Upper Cretaceous rocks of the Black Hills region, even though the thin sections examined were selected in the belief that they were representative. This uncertainty will remain until many more than 30 thin sections have been examined. Also, parts of the shale that show no laminations may have accumulated much more slowly than the others. However, with present information, it seems that the entire discrepancy can not be explained in this way, because in general rock types that show the thickest laminations, and hence seem to have accumulated most rapidly, show the most evidence of destruction of laminations (pl. 5, A) and also the largest proportion of

obscurely laminated or entirely unlaminated shale, whereas the most thinly laminated rocks tend to be the most distinctly laminated. (See pp. 51–52, 53.)

Some scraps of evidence favor one of the other two explanations. The total of about 2,500,000 laminations found by Stamp⁴⁹ in the Oligocene and lower Miocene deposits of Burma, like that of the Upper Cretaceous laminations, is only about one-tenth the number of years estimated by Barrell⁵⁰ as the time during which the rocks accumulated. And Barrell, when defining diastems as minor but very common stratigraphic breaks caused by downward oscillation of wave base,⁵¹ seems from the illustrative examples he cited to have thought that the aggregate time value of stratigraphic breaks might be five or ten times that recorded in the remaining deposits.⁵² Stamp's results seem to accord with the writer's in suggesting that the estimates based on lead-uranium ratios or the proportions assigned to certain periods may be too long or that inconspicuous stratigraphic breaks may be very common. Barrell's theoretical conclusions favor the latter possibility.

CONDITIONS OF DEPOSITION INDICATED BY LAMINATIONS

Whether or not the laminations are annual, they furnish a clue to some of the conditions of deposition. Their distinctness or degree of preservation is probably a rough measure of the quietness of the water in which the sediments accumulated. Very slight stirring would destroy thin laminations in previously deposited clay; and the disturbed fine particles would be redeposited as a mixture with coarser material that came in later.⁵³ Distinct laminations would be formed and preserved only below the depth at which waves could move the very fine sediments; and therefore distinctly laminated sediments probably accumulated in deep water or at times of mild climate and gentle winds. The distinctness of laminations would probably depend also on the salinity of the water⁵⁴ and the climatic contrasts between the seasons of the year, but, as the Upper Cretaceous rocks of the Black Hills region are nearly all marine and as the available evidence indicates a mild but distinctly seasonal climate in the Black Hills region during Upper Cretaceous time, these conditions were fulfilled here.

On applying this test to the samples studied, it appears that the fine-grained noncalcareous shales with the organic type of laminations accumulated in deep water or at times of gentle winds. The coarser silt

⁴⁹ Stamp, L. D., *Seasonal rhythm in the Tertiary sediments of Burma*: Geol. Mag., vol. 62, pp. 526–527, 1925.

⁵⁰ Barrell, Joseph, *op. cit.*, p. 884.

⁵¹ *Idem*, pp. 748, 785–795.

⁵² *Idem*, pp. 796–797, 807, fig. 5.

⁵³ Barrell, Joseph, *Criteria for the recognition of ancient delta deposits*: Geol. Soc. America Bull., vol. 23, pp. 425–427, 1912.

⁵⁴ Sauramo, Matti, *op. cit.* pp. 90, 92–93, 97–98, 110.

and clay layers are equally distinct, but, in view of the larger waves or shallower depths necessary to account for the disturbance of the coarser grains, it seems that the distinctly laminated finer-grained rocks probably accumulated in deeper waters than the equally well laminated coarser rocks.

This test also indicates that the mudstones in the upper part of the Pierre shale and the calcareous marls of the Niobrara formation accumulated in relatively shallow water or at times when wave base extended deeper than usual, for they show little or no lamination. Loss of laminations in the upper Pierre mudstones by wave disturbance is further indicated by the minute contemporaneous brecciation shown by several specimens examined. (See pl. 5, A.) The suggestion that the calcareous marls of the Niobrara formation may have accumulated in relatively shallow water accords with the greater abundance of mollusk shells in the calcareous rocks (see pp. 12, 53) and with the greater solubility of calcium carbonate in cold (deep) water than in warm (shallow) water.⁵⁵

If the different kinds of paired laminations were formed during equal time intervals (whether or not these time intervals were years), the rocks with thicker pairs of laminations accumulated more rapidly than the rocks with thinner ones. Hence, the relative thicknesses of the pairs of laminations in different samples may indicate the relative rates of accumulation of different lithologic types and formations as a whole. However, this criterion must be used cautiously, for, as Barrell⁵⁶ pointed out, the more rapid the accumulation of individual beds the longer the alternating periods of nondeposition may be. Nevertheless, it is interesting to note that the Mowry shale, which has the thinnest pairs of laminations observed, affords independent evidence that it accumulated much more slowly⁵⁷ than other formations of the region. If the thickness of the pairs of laminations indicates the relative rates of accumulation of the different rock types and formations, the sandy shales accumulated most rapidly, the calcareous marls probably somewhat less rapidly, and the fine-grained organic shales most slowly. (See also pp. 13, 53.)

SUMMARY

Microscopic examination reveals alternate laminations that average about 0.2 millimeter in thickness

⁵⁵ Wells, R. C., The solubility of calcite in water in contact with the atmosphere and its variation with temperature: *Washington Acad. Sci. Jour.*, vol. 5, pp. 617-622, 1915. Johnston, John, and Williamson, E. D., The rôle of inorganic agencies in the deposition of calcium carbonate: *Jour. Geology*, vol. 24, pp. 729-750, 1916. Wells, R. C., New determinations of carbon dioxide in water of the Gulf of Mexico: *U. S. Geol. Survey Prof. Paper 120*, p. 11, 1919. Rubey, W. W., Origin of the siliceous Mowry shale of the Black Hills region: *U. S. Geol. Survey Prof. Paper 154*, pp. 164-165, 1928.

⁵⁶ Barrell, Joseph, *op. cit.*, p. 801.

⁵⁷ Rubey, W. W., *op. cit.*, pp. 156, 167-169.

in marine shale of many Upper Cretaceous formations in the Black Hills region. These laminations are of different kinds, marked by alternations of three types—(1) coarse and fine particles, (2) much and little organic matter, and (3) calcium carbonate and silt. Examples intermediate between types 1 and 2 form a gradational series in which those pairs of laminations made by alternations of particle size are the thicker and those made by alternations of organic content the thinner. This gradation indicates that at least these two kinds of alternate laminations were formed by the same or a closely related process and during approximately equal time intervals.

Storms or floods might have caused the alternations of coarse and fine particles, but the pairs of laminations marked by varying content of organic matter and of calcium carbonate seem to call for recurrent cycles of organic growth or of changes in temperature. The fact that thin laminations have been preserved indicates that sporadic storms rarely disturbed the sea floor, and the regularity of the alternations suggests that the cause, whether storms or not, recurred periodically. Either seasonal changes in temperature, rainfall, and food supply of organisms or periodic shifts of marine currents (warm and cold) probably afford the simplest explanation of all three kinds of alternations; and of these two possibilities seasonal changes appear the more likely.

It is conceivable that annual layers might have formed in the Upper Cretaceous rocks of the Black Hills region, for fossil wood and other evidence indicate that the climate was seasonal; and flocculation, which, according to some geologists, might prevent the separation of sand and clay into coarse and fine layers, probably would not prevent the formation of layers marked by alternations of either organic matter or calcium carbonate. Once formed, thin layers might have been preserved, for the deepest part of the Upper Cretaceous interior sea probably lay near the present Black Hills; also, wave action may not have extended as deep during the widespread equable climates of Upper Cretaceous time as it does to-day.

The hypothesis that the laminations are annual is tested roughly by comparing the thickness of the observed laminations with the thickness that annual layers might be expected to have. Varves in glacial deposits of other regions are commonly much thicker, but many varves in lake deposits and in some marine rocks are of about the same thickness as these laminations. Estimates of the expected thickness of annual layers in the Upper Cretaceous rocks near the Black Hills also appear to support the hypothesis. Three methods of estimation—(1) the rhythmic alternations in Upper Cretaceous rocks in eastern Colorado, which Gilbert suggested were formed during precession cycles, (2) the probable area of land draining into the

Upper Cretaceous sea and the present rate of erosion in the Mississippi drainage basin, (3) the total thickness of Upper Cretaceous rocks in the region divided by Barrell's estimate of the number of years in the Upper Cretaceous epoch—all indicate annual layers only slightly thinner than the observed laminations. A modification of the third method, in which the total thickness of Upper Cretaceous rocks is divided by estimates of Upper Cretaceous time based on the amount of salt in the ocean, indicates annual layers somewhat thicker than the observed laminations.

Thus the laminations are of about the right thickness to be annual; they were not caused by daily variations or by cycles several thousand years long. However, this is no proof that they are annual, for they might have formed every few months or years. In fact, more detailed comparisons (*a*) of the rate of eastward transgression of the Upper Cretaceous sea (calculated from the number of laminations and the distance of the transgression) with the probable average rate of strand-line movements to-day, and (*b*) of the length of Upper Cretaceous time indicated by the laminations with that estimated by Barrell, suggest that they may have formed every few years. Yet these two apparent discrepancies might be explained equally well (*a*) if the sea advanced rapidly then because it came in over a relatively flat surface, and (*b*) if Barrell's estimate of Upper Cretaceous time is too long or if there are many inconspicuous unconformities or diastems in the stratigraphic section.

Whether or not the laminations are annual, they suggest some of the conditions of deposition. The degree of preservation of the laminations indicates that the fine-grained organic shales accumulated in deep water or at times of gentle winds. If the laminations of different kinds formed during equal time intervals, their relative thicknesses indicate that, in general, the sandier shales and calcareous marls accumulated more rapidly and the finer grained and more organic shales more slowly.

COORDINATION OF CONCLUSIONS

Several of the conclusions reached in the separate discussions of different lithologic characteristics of these rocks overlap to a certain extent. Marshaled together, these overlapping conclusions acquire a higher degree of probability and the evidence on which they are based takes on a somewhat broader significance. Purely physical problems, such as the part played by adsorbed surface films in the behavior of argillaceous rocks, being neglected as outside the scope of this paper, the points of geologic interest, upon which several lines of evidence seem to converge, fall into three groups—the conditions of deposition of the sediments, the manner in which the rocks were later deformed, the bearing of these factors

upon a proper appraisal of the oil and gas possibilities of the region.

Conditions of deposition.—Two features of the environment of marine sediments that influence to a considerable degree the nature of the sediments are the depth of water and the rate of deposition. In a general way, the temperature, agitation by waves, oxygen content, and habitability (which depends upon the availability of oxygen, light, and food) of sea water decrease with increasing depth. A consideration of the content of calcium carbonate, organic matter, pyrite, and fossils in the different specimens led to the conclusion that the more calcareous rocks probably accumulated in relatively warm water, in which many organisms lived and died. From the general relation of temperature and habitability to depth and from analogy with blue muds that are forming to-day, it seems that these calcareous rocks probably accumulated in relatively shallow water. This conclusion accords with that drawn from a study of the bedding laminations, for the noncalcareous shales are distinctly laminated, and the more calcareous rocks show evidence of loss of bedding by wave disturbance. In fact, it is possible that difference of depth and degree of wave agitation may in part account for the lack of sorting in the noncalcareous shales and the somewhat better sorting of the calcareous rocks.

The theoretical consideration of the conditions that might favor the preservation of calcium carbonate, organic matter, and pyrite together, supplemented by the analogy with present-day blue muds, and the empirical observation of the thickness of bedding laminations in the different rock types indicate that the organic and inorganic matter in the more calcareous rocks probably accumulated more rapidly than that in the fine-grained noncalcareous shales. The inverse relation between rate of deposition and depth of water that is thus suggested by these studies is not surprising. Areas where the water is shallow are commonly near the source or land from which sediment and food are carried into the sea. However, the relation is obviously not a general one that applies to all environments where marine sediments are laid down. The very fact that more detritus is available near shore means that a larger proportion of noncalcareous sediment is deposited there. And this sediment, unless the near-shore areas are sinking rapidly, is repeatedly eroded and redistributed by waves, so that the average rate of accumulation near shore is greatly reduced. It may well be that from deep water up to some unknown depth or some unknown distance from shore decreasing depth and increasing rate of sedimentation tend to give progressively more calcareous sediments, but that at still shallower depths or nearer shore this relation no longer holds.

Deformation of the rocks.—The loss of porosity with increasing depth of burial and steepness of dip, which is shown by these samples, implies a decrease of rock volume and an internal deformation of the rock mass. The fissility of the shales and the aggregate optical orientation of mineral particles also seem to show that there has been internal rearrangement of particles as the result of burial. Likewise, field evidence that shale beds have been squeezed thinner where steeply folded indicates loss of volume as the beds are tilted.

The apparent relations of the porosity to the depth of burial and to the steepness of dip, together with the partial confirmation of these relations from other lines of evidence, lead to several important corollaries. Compaction, or loss of porosity and increase of density, by burial is a continuous but decreasingly important process. Compaction by tilting indicates that horizontal compression deformed the rocks, that their apparent stratigraphic thickness depends upon the degree of folding, and that large volumes of water were squeezed out of them during folding. Although these rocks have yielded under load and pressure like incompetent members, by flattening and by bending into similar folds, they have not undergone true plastic or constant-volume deformation. Under both loading and tilting they have been thinned stratigraphically but not appreciably lengthened parallel to the bedding. However, at lesser and greater loads and pressures, these relations probably would not hold.

Occurrence of oil and gas.—Several lines of evidence seem to bear upon the oil and gas possibilities of these rocks. The percentages of chloroform-soluble organic matter in the samples apparently vary with the stratigraphic position, with the total amount of organic matter, with the composition of the organic matter, and with either the degree of deformation or the porosity of the rocks—it is impossible to say which, because the porosity depends in turn upon the degree of deformation. Perhaps all these relations might be deduced from theoretical considerations. Nevertheless it is worth noting that the

suggested possibility that the deformation of the rocks may be an important factor in the generation of petroleum seems to accord with the results of drilling, for nearly all occurrences of oil in the Black Hills region are closely associated with steep dips and faulting. Porosity seems related both to the angle of dip or the shear and to the depth of burial or the load, varying inversely with the deformation. The percentage of soluble bitumens also seems to increase with the dip or shear; but, on the other hand, it seems to decrease with the depth or load. This observation suggests the possibility that there is a fundamental difference between the effects upon organic matter of straight compression and of sliding shear—in fact, this difference is a possible explanation of the contradictory results of published laboratory experiments.

Whether or not petroleum is derived from organic matter by folding, it must still, in order to form oil pools, migrate from the source rocks and accumulate in adjacent reservoir rocks or sandstones. The very fact that soluble bitumens are more abundant in steeply dipping than in horizontal beds might mean that the soluble bitumens remain where they are formed and do not migrate into interlaminated sandstones. However, the decrease of pore space with increase of dip implies that fluid was squeezed out of the rock, either upward along fissures or laterally through interlaminated sandstone beds, and presumably some of the more liquid hydrocarbons would be carried away with this fluid; that is, folding might not only form the petroleum but cause some of it to migrate from the source rocks into adjacent sandstones.

This suggestion, based on the study of a few specimens, is, of course, merely a possibility that must be tested by other investigations. This assumed effect of deformation on the generation and migration of petroleum and the more definitely observed stratigraphic distribution of chloroform-soluble organic matter in the rocks suggest possible applications or guides in the search for new oil fields in the Black Hills region.

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